

## **Europäisches Patentamt**

**European Patent Office** 

Office européen des brevets



EP 0 924 690 A2 (11)

(12)

## **EUROPEAN PATENT APPLICATION**

(43) Date of publication:

23.06.1999 Bulletin 1999/25

(21) Application number: 98310175.9

(22) Date of filing: 11.12.1998

(84) Designated Contracting States:

AT BE CH CY DE DK ES FI FR GB GR IE IT LI LU MC NL PT SE

**Designated Extension States:** 

**AL LT LV MK RO SI** 

(30) Priority: 12.12.1997 JP 36270197

(71) Applicant: TODA KOGYO CORP. Hiroshima-shi Hiroshima-ken (JP)

(72) Inventors:

· Hayashi, Kazuyuki Hiroshima-shi, Hiroshima-ken (JP) · Tanaka, Yasuyuki Onoda-shi, Yamaguchi-ken (JP)

(51) Int. Cl.<sup>6</sup>: **G11B 5/712** 

 Iwasaki, Keisuke Hiroshima-shi, Hiroshima-ken (JP)

 Morii, Hiroko Hiroshima-shi, iroshima-ken (JP)

(74) Representative:

Woods, Geoffrey Corlett J.A. KEMP & CO. 14 South Square Gray's Inn London WC1R 5LX (GB)

#### Magnetic recording medium and substrate therefor (54)

- (57)A magnetic recording medium comprises:
  - (a) a non-magnetic base film;
  - (b) a non-magnetic undercoat layer on said nonmagnetic base film, comprising a binder resin and non-magnetic acicular black iron-based composite particles; and
  - (c) a magnetic coating film comprising a binder resin and magnetic particles, comprising
  - acicular hematite or iron oxide hydroxide particles having an average major axis diameter of from 0.02 to 0.30 µm,
  - a coating layer on the surface of said acicular hematite or iron oxide hydroxide particles, comprising at least one organosilicon compound selected from:
    - (1) organosilane compounds obtainable by drying or heat-treating alkoxysilane compounds,
    - (2) polysiloxanes or modified polysiloxanes, and
    - (3) fluoroalkyl organosilane compounds obtainable by drying or heat-treating a fluoroalkylsilane compounds, and
  - from 1 to 20 parts by weight, per 100 parts by weight of said acicular hematite or iron oxide hydroxide particles, of carbon black particles

which have a particle size of from 0.002 to 0.05  $\mu m$  and which are adhered to at least a part of said coating layer.

## Description

35

**[0001]** The present invention relates to a magnetic recording medium and a substrate therefor, and more particularly, to a magnetic recording medium comprising a non-magnetic undercoat layer having a smooth surface, a high strength and a lower light transmittance and a low electric resistance even at a small carbon black content, and a substrate for the magnetic recording medium.

**[0002]** With a development of miniaturized and lightweight video or audio magnetic recording and reproducing apparatuses for long-time recording, magnetic recording media such as a magnetic tape and magnetic disk have been increasingly and strongly required to have a higher performance, namely, a higher recording density, higher output characteristic, in particular, an improved frequency characteristic and a lower noise level.

**[0003]** A reduction in the thickness of a magnetic, recording layer is described. Video tapes have recently been required more and more to have a higher picture quality, and the frequencies of carrier signals recorded in recent video tapes are higher than those recorded in conventional video tapes. In other words, the signals in the short-wave region have come to be used, and as a result, the magnetization depth from the surface of a magnetic tape has come to be remarkably small.

[0004] With respect to short wavelength signals, a reduction in the thickness of a magnetic recording layer is also strongly demanded in order to improve the high output characteristics, especially, the S/N ratio of a magnetic recording medium. This fact is described, for example, on page 312 of <u>Development of Magnetic Materials and Technique for High Dispersion of Magnetic Powder</u>, published by Sogo Gijutsu Center Co., Ltd. (1982), "• • the conditions for high-density recording in a coated-layer type tape are that the noise level is low with respect to signals having a short wavelength and that the high output characteristics are maintained. To satisfy these conditions, it is necessary that the tape has large coercive force Hc and residual magnetisation Br, • • and the coating film has a smaller thickness. • • • ".

[0005] Development of a thinner film for a magnetic recording layer has caused some problems.

[0006] Firstly, it is necessary to make a magnetic recording layer smooth and to eliminate the non-uniformity of thickness. As well known, in order to obtain a smooth magnetic recording layer having a uniform thickness, the surface of the base film must also be smooth. This fact is described on pages 180 and 181 of Materials for Synthetic Technology-Causes of Friction and Abrasion of Magnetic Tape and Head Running System and Measures for Solving the Problem (hereinunder referred to as "Materials for Synthetic Technology" (1987), published by the Publishing Department of Technology Information Center, "• • • the surface roughness of a hardened magnetic coating film depends on the surface roughness of the base film (back surface roughness) so largely as to be approximately proportional, • • •, since the magnetic coating film is formed on the base film, the more smooth the surface of the base film is, the more uniform and larger head output is obtained and the more the S/N ratio is improved."

[0007] Secondly, there has been caused a problem in the strength of a base film with a tendency of the reduction in the thickness of the base film in response to the demand for a thinner magnetic coating film. This fact is described, for example, on page 77 of the above-described Development of Magnetic Materials and Technique for High Dispersion of Magnetic Powder, "• • Higher recording density is a large problem assigned t the present magnetic tape. This is important in order to shorten the length of the tape so as to miniaturize the size of a cassette and to enable long-time recording. For this purpose, it is necessary to reduce the thickness of a base film • • • . With the tendency of reduction in the film thickness, the stiffness of the tape also reduces to such an extent as to make smooth travel in a recorder difficult. Therefore, improvement of the stiffness of a video tape both in the machine direction and in the transverse direction is now strongly demanded. • • • "

[0008] The end portion of a magnetic recording medium such as a magnetic tape, especially, a video tape is judged by detecting a portion of the magnetic recording medium at which the light transmittance is large by a video deck. If the light transmittance of the whole part of a magnetic recording layer is made large by the production of a thinner magnetic recording medium or the ultrafine magnetic particles dispersed in the magnetic recording layer, it is difficult to detect the portion having a large light transmittance by a video deck. For reducing the light transmittance of the whole part of a magnetic recording layer, carbon black or the like is added to the magnetic recording layer. It is, therefore, essential to add carbon black or the like to a magnetic recording layer in the present video tapes.

**[0009]** However, addition of a large amount of non-magnetic particles such as carbon black impairs not only the enhancement of the magnetic recording density but also the development of a thinner recording layer. In order to reduce the magnetization depth from the surface of the magnetic tape and to produce a thinner magnetic recording layer, it is strongly demanded to reduce, as much as possible, the quantity of non-magnetic particles such as carbon black which are added to a magnetic recording layer.

**[0010]** Consequently, it has been strongly demanded to provide a magnetic recording medium capable of exhibiting a low light transmittance even when the amount of carbon black added to a magnetic recording layer thereof is reduced as low as possible. From this viewpoint, it has been strongly required to essentially improve properties of a substrate therefor

[0011] Further, in order to reduce not only the above-mentioned light transmittance of the magnetic recording medium

but also an electric resistance thereof, there has been hitherto proposed a method of adding carbon black to the magnetic recording layer.

[0012] The conventional magnetic recording medium to which carbon black is added, is described in detail below.

[0013] When a magnetic recording medium has a high surface resistivity, an electrostatic charge thereon tends to be increased, so that cut chips of the magnetic recording medium and dirt or dusts are attached onto the surface of the magnetic recording medium upon production or use thereof, and as a result, such a problem that the number of "dropout" becomes increased, is caused.

**[0014]** In order to decrease the surface resistivity of the magnetic recording medium to about  $10^8 \Omega$ /sq, a conductive compound such as carbon black has been ordinarily added to the magnetic recording layer in an amount of not less than about 5 parts by weight based on 100 parts of magnetic particles contained in the magnetic recording layer.

**[0015]** However, when the amount of carbon black added or the like which cannot contribute to improvement in magnetic properties of the magnetic recording layer, is increased, the magnetic recording medium has been deteriorated in signal recording property as described above, resulting in inhibiting the magnetic recording layer from being thinned.

[0016] With the reduction in thickness of the magnetic recording layer or the base film therefor, various attempts have been conducted in order to improve properties of a substrate on which the magnetic recording layer is formed, thereby enhancing a surface smoothness and a strength of the magnetic recording medium. In this regard, there has been proposed a method of forming on a base film, at least one undercoat layer composed of a binder resin and non-magnetic particles dispersed in the binder resin and containing iron as a main component, such as acicular hematite particles or acicular iron oxide hydroxide particles (hereinafter referred to merely as "non-magnetic undercoat layer"), and such a method has been already put into practice (Japanese Patent Publication (KOKOKU) No. 6-93297(1994), Japanese Patent Application Laid-open (KOKAI) Nos. 62-159338(1987), 63-187418(1988), 4-167225(1992), 4-325915(1992), 5-73882(1993), 5-182177(1993), 5-347017(1993) and 6-60362(1994), and the like).

[0017] It is known to use as the non-magnetic particles for non-magnetic undercoat layer, non-magnetic particles which surfaces are coated with hydroxides of aluminum, oxides of aluminum, hydroxides of silicon, oxides of silicon or a mixture thereof in order to improve the dispersibility thereof in vehicles, thereby enhancing a surface smoothness and a strength of the obtained non-magnetic substrate (Japanese Patent Nos. 2,571,350 and 2,582,051, and Japanese Patent Application Laid-open (KOKAI) Nos. 6-60362(1994), 9-22524(1997) and 9-27117(1997) or the like).

[0018] Also, it is known that in order to decrease an amount of carbon black added to the magnetic recording layer and reduce a light transmittance of the magnetic recording medium, blackish brown acicular hematite particles or blackish brown acicular ferric oxide hydroxide particles are used as the non-magnetic particles for non-magnetic undercoat layer (Japanese Patent Application Laid-open (KOKAI) Nos. 7-66020(1995) and 8-259237(1996)).

**[0019]** Further, it is known that in order to reduce an electric resistance of the magnetic recording medium, a mixture of non-magnetic iron oxide particles and carbon black particles is used as the non-magnetic particles for non-magnetic undercoat layer (Japanese Patent Application Laid-open (KOKAI) Nos. 1-213822(1989), 1-300419(1989), 6-236542(1994) and 9-297911(1997) or the like).

[0020] With the reduction in thickness of not only the magnetic recording layer but also the base film, it has been most demanded to provide such a magnetic recording medium having a more smooth surface, a high strength, and a lower light transmittance and a lower electric resistance even when the amount of carbon black in the magnetic recording layer is reduced as low as possible. However, at present, magnetic recording media capable of satisfying all of such requirements have not been obtained yet.

[0021] Namely, in the case of the above-mentioned conventional substrates produced by using as the non-magnetic particles for non-magnetic undercoat layer, acicular hematite particles, acicular iron oxide hydroxide particles, acicular hematite particles coated with hydroxides of aluminum or the like, or acicular iron oxide hydroxide particles coated with hydroxides of aluminum or the like, although surface smoothness and high strength of the substrate using these particles as non-magnetic particles are satisfied, it is difficult to reduce the light transmittance due to dark red or yellowish brown color of the non-magnetic particles. In addition, the non-magnetic substrate exhibit a surface resistivity as large as not less than about  $10^{13} \Omega/\text{sq}$ .

**[0022]** Further, in the case of the above-mentioned substrate using as non-magnetic particles for non-magnetic undercoat layer, blackish brown acicular hematite particles or blackish brown acicular iron oxide hydroxide particles, the obtained non-magnetic undercoat layer can show a higher degree of blackness as compared to those using the dark red acicular hematite particles or the yellowish brown acicular iron oxide hydroxide particles, so that it becomes possible to reduce a light transmittance of the substrate. However, the reduction of light transmittance is still insufficient. In addition, the surface resistivity of the non-magnetic substrate is as large as about  $10^{12} \Omega$ /sq.

[0023] In the case of the non-magnetic particles described in the above-mentioned Japanese Patent Application Laidopen (KOKAI) Nos. 1-213822(1989), 1-300419(1989) and 9-297911(1997), there have been used the mixture of non-magnetic iron oxide particles and carbon black particles in which the carbon black particles are added in an amount of not less than 25 parts by weight based on 100 parts by weight of the non-magnetic iron oxide particles. Therefore, due to the fact that the carbon black particles which show the largest degree of blackness among various black pigments,

are used in such a large amount, it is possible to obtain a non-magnetic substrate having a low light transmittance and a low electric resistance. However, it has been difficult to disperse the carbon black particles in vehicles, since the carbon black particles are fine particles having such a small average major axis diameter as about 0.002 to about 0.05 μm, a large specific surface area and a deteriorated solvent wettability, thereby failing to obtain a non-magnetic substrate having a smooth surface and a high strength. In addition, the carbon black particles have a bulk density as low as about 0.1 g/cm<sup>3</sup> and, therefore, the carbon black particles are bulky particles, resulting in deteriorated handing property and workability. Further, with respect to the carbon black particles, many problems concerning safety or hygiene such as carcinogenesis have been pointed out.

[0024] Thus, as the amount of the carbon black particles added to the non-magnetic substrate is increased, the light transmittance of the obtained non-magnetic substrate tends to become small. However, when the carbon black particles are used in a large amount, it becomes more difficult to disperse the particles in vehicles, resulting in deteriorated workability. Further, the use of a large amount of the carbon black particles are disadvantageous in view of safety and hygiene.

[0025] In the above-mentioned Japanese Patent Application Laid-open (KOKAI) No. 6-236542(1994), there have been described such non-magnetic particles mixture of non-magnetic iron oxide particles and carbon black having a specific structure in which the carbon black is used in an amount of 1 to 17.6 parts by weight based on 100 parts by weight of the non-magnetic iron oxide particles. By using such specific carbon black having a high conductivity, the electric resistance of the obtained non-magnetic substrate can be reduced even at a small carbon black content. However, since the amount of carbon black used is small, it is difficult to reduce a light transmittance of the non-magnetic substrate.

**[0026]** In consequence, it has been strongly demanded to provide a magnetic recording medium and a non-magnetic substrate therefor which can exhibit a smooth surface, a high strength, and a lower light transmittance and a lower electric resistance even at a small carbon black content.

[0027] As a result of the present inventors' earnest studies, it has been found that by using as non-magnetic particles contained in a non-magnetic undercoat layer, non-magnetic acicular black iron-based composite particles comprising:

acicular hematite particles or acicular iron oxide hydroxide particles having an average major axis diameter of 0.02 to 0.30 µm;

a coating layer formed on surface of said acicular hematite particle or acicular iron oxide hydroxide particle, comprising at least one organosilicon compound selected from the group consisting of:

- (1) organosilane compounds obtained by drying or heat-treating an alkoxysilane compounds,
- (2) polysiloxanes or modified polysiloxanes, and
- (3) fluoroalkyl organosilane compounds obtained by drying or heat-treating a fluoroalkylsilane compounds; and

carbon black fine particles having a particle size of 0.002 to  $0.05~\mu m$  adhered on at least a part of said coating layer, the amount of said carbon black fine particles adhered being 1 to 20 parts by weight based on 100 parts by weight of said acicular hematite particles or acicular iron oxide hydroxide particles,

the obtained non-magnetic substrate can exhibit a more excellent smooth surface, a high strength, and a lower light transmittance and a lower electric resistance even at a small carbon black content. The present invention has been attained on the basis of this finding.

[0028] It is an object of the present invention to provide a non-magnetic substrate for a magnetic recording medium which can show a more smooth surface, a high strength, and a lower light transmittance and a lower electric resistance even at a small carbon black content.

**[0029]** It is another object of the present invention to provide a magnetic recording medium having a non-magnetic undercoat layer which can show a more smooth surface, a high strength, and a lower light transmittance and a lower electric resistance even at a small carbon black content.

[0030] To accomplish the aims, in a first aspect of the present invention, there are provided a magnetic recording medium comprising:

a non-magnetic base film;

30

35

40

55

a non-magnetic undercoat layer formed on said non-magnetic base film, comprising a binder resin and non-magnetic acicular black iron-based composite particles; and

a magnetic coating film comprising a binder resin and magnetic particles,

said non-magnetic acicular black iron-based composite particles comprising:

acicular hematite particles or acicular iron oxide hydroxide particles having an average major axis diameter of 0.02

to 0.30 µm;

5

20

25

30

35

40

45

50

55

a coating layer formed on surface of said acicular hematite particle or acicular iron oxide hydroxide particle, comprising at least one organosilicon compound selected from the group consisting of:

- (1) organosilane compounds obtained by drying or heat-treating an alkoxysilane compounds,
- (2) polysiloxanes or modified polysiloxanes, and
- (3) fluoroalkyl organosilane compounds obtained by drying or heat-treating a fluoroalkylsilane compounds; and
- carbon black fine particles having a particle size of 0.002 to 0.05 μm, adhered on at least a part of said coating layer,

the amount of said carbon black fine particles adhered being 1 to 20 parts by weight based on 100 parts by weight of said acicular hematite particles or acicular iron oxide hydroxide particles.

15 [0031] In a second aspect of the present invention, there are provided a magnetic recording medium comprising:

a non-magnetic base film;

a non-magnetic undercoat layer formed on said non-magnetic base film, comprising a binder resin and non-magnetic acicular black iron-based composite particles; and

a magnetic coating film comprising a binder resin and magnetic particles,

said non-magnetic acicular black iron-based composite particles comprising:

acicular hematite particles or acicular iron oxide hydroxide particles having an average major axis diameter of 0.02 to 0.30 µm;

a coating layer formed on surface of said acicular hematite particle or acicular iron oxide hydroxide particle, comprising at least one organosilicon compound selected from the group consisting of:

- (1) organosilane compounds obtained by drying or heat-treating an alkoxysilane compounds,
- (2) polysiloxanes or modified polysiloxanes, and
- (3) fluoroalkyl organosilane compounds obtained by drying or heat-treating a fluoroalkylsilane compounds; and

carbon black fine particles having a particle size of 0.002 to 0.05  $\mu$ m, adhered on at least a part of said coating layer,

the amount of said carbon black fine particles adhered being 1 to 20 parts by weight based on 100 parts by weight of said acicular hematite particles or acicular iron oxide hydroxide particles,

said acicular hematite particles or acicular iron oxide hydroxide particles being particles having a coat formed on at least a part of the surface of said acicular hematite particles or acicular iron oxide hydroxide particles and comprising at least one compound selected from the group consisting of hydroxides of aluminum, oxides of aluminum, hydroxides of silicon and oxides of silicon in an amount of 0.01 to 50 % by weight (calculated as Al or SiO<sub>2</sub>) based on the total weight of the acicular hematite particles or acicular iron oxide hydroxide particles.

[0032] In a third aspect of the present invention, there are provided a magnetic recording medium comprising:

a non-magnetic base film;

a non-magnetic undercoat layer formed on said non-magnetic base film, comprising a binder resin and non-magnetic acicular black iron-based composite particles; and

a magnetic coating film comprising a binder resin and magnetic particles,

said non-magnetic acicular black iron-based composite particles comprising:

acicular manganese-containing hematite particles or acicular manganese-containing iron oxide hydroxide particles having an average major axis diameter of 0.02 to 0.30 µm;

a coating layer formed on surface of said acicular manganese-containing hematite particle or acicular manganese-containing iron oxide hydroxide particle, comprising at least one organosilicon compound selected from the group consisting of:

- (1) organosilane compounds obtained by drying or heat-treating an alkoxysilane compounds,
- (2) polysiloxanes or modified polysiloxanes, and
- (3) fluoroalkyl organosilane compounds obtained by drying or heat-treating a fluoroalkylsilane compounds; and

carbon black fine particles having a particle size of 0.002 to  $0.05~\mu m$ , adhered on at least a part of said coating layer.

the amount of said carbon black fine particles adhered being 1 to 20 parts by weight based on 100 parts by weight of said acicular manganese-containing hematite particles or acicular manganese-containing iron oxide hydroxide particles.

[0033] In a fourth aspect of the present invention, there are provided a magnetic recording medium comprising:

a non-magnetic base film;

5

10

15

20

30

35

50

55

a non-magnetic undercoat layer formed on said non-magnetic base film, comprising a binder resin and non-magnetic acicular black iron-based composite particles; and

a magnetic coating film comprising a binder resin and magnetic particles,

said non-magnetic acicular black iron-based composite particles comprising:

acicular manganese-containing hematite particles or acicular manganese-containing iron oxide hydroxide particles having an average major axis diameter of 0.02 to 0.30 µm;

a coating layer formed on surface of said acicular manganese-containing hematite particle or acicular manganese-containing iron oxide hydroxide particle, comprising at least one organosilicon compound selected from the group consisting of:

- (1) organosilane compounds obtained by drying or heat-treating an alkoxysilane compounds,
- (2) polysiloxanes or modified polysiloxanes, and
- (3) fluoroalkyl organosilane compounds obtained by drying or heat-treating a fluoroalkylsilane compounds; and
- carbon black fine particles having a particle size of 0.002 to 0.05 μm, adhered on at least a part of said coating layer.

the amount of said carbon black fine particles adhered being 1 to 20 parts by weight based on 100 parts by weight of said acicular manganese-containing hematite particles or acicular manganese-containing iron oxide hydroxide particles,

said acicular manganese-containing hematite particles or acicular manganese-containing iron oxide hydroxide particles being particles having a coat formed on at least a part of the surface of said acicular manganese-containing hematite particles or acicular manganese-containing iron oxide hydroxide particles and comprising at least one compound selected from the group consisting of hydroxides of aluminum, oxides of aluminum, hydroxides of silicon and oxides of silicon in an amount of 0.01 to 50 % by weight (calculated as Al or SiO<sub>2</sub>) based on the total weight of the acicular manganese-containing hematite particles or acicular manganese-containing iron oxide hydroxide particles.

[0034] In a fifth aspect of the present invention, there are provided a non-magnetic substrate comprising:

- a non-magnetic base film; and a non-magnetic undercoat layer formed on said non-magnetic base film, comprising a binder resin and non-magnetic acicular black iron-based composite particles,
  - said non-magnetic acicular black iron-based composite particles comprising:
  - acicular hematite particles or acicular iron oxide hydroxide particles having an average major axis diameter of 0.02 to  $0.30 \mu m$ ;
- a coating layer formed on surface of said acicular hematite particle or acicular iron oxide hydroxide particle, comprising at least one organosilicon compound selected from the group consisting of:
  - (1) organosilane compounds obtained by drying or heat-treating an alkoxysilane compounds,
  - (2) polysiloxanes or modified polysiloxanes, and
  - (3) fluoroalkyl organosilane compounds obtained by drying or heat-treating a fluoroalkylsilane compounds; and

carbon black fine particles having a particle size of 0.002 to  $0.05~\mu m$ , adhered on at least a part of said coating layer.

the amount of said carbon black fine particles adhered being 1 to 20 parts by weight based on 100 parts by weight of said acicular hematite particles or acicular iron oxide hydroxide particles.

[0035] In a sixth aspect of the present invention, there are provided a non-magnetic substrate comprising:

a non-magnetic base film; and

5

10

20

30

35

40

45

55

a non-magnetic undercoat layer formed on said non-magnetic base film, comprising a binder resin and non-magnetic acicular black iron-based composite particles,

said non-magnetic acicular black iron-based composite particles comprising:

- acicular hematite particles or acicular iron oxide hydroxide particles having an average major axis diameter of 0.02 to 0.30 μm;
- a coating layer formed on surface of said acicular hematite particle or acicular iron oxide hydroxide particle, comprising at least one organosilicon compound selected from the group consisting of:
  - (1) organosilane compounds obtained by drying or heat-treating an alkoxysilane compounds,
  - (2) polysiloxanes or modified polysiloxanes, and
  - (3) fluoroalkyl organosilane compounds obtained by drying or heat-treating a fluoroalkylsilane compounds; and
- carbon black fine particles having a particle size of 0.002 to 0.05  $\mu$ m, adhered on at least a part of said coating layer.

the amount of said carbon black fine particles adhered being 1 to 20 parts by weight based on 100 parts by weight of said acicular hematite particles or acicular iron oxide hydroxide particles,

said acicular hematite particles or acicular iron oxide hydroxide particles being particles having a coat formed on at least a part of the surface of said acicular hematite particles or acicular iron oxide hydroxide particles and comprising at least one compound selected from the group consisting of hydroxides of aluminum, oxides of aluminum, hydroxides of silicon and oxides of silicon in an amount of 0.01 to 50 % by weight (calculated as Al or SiO<sub>2</sub>) based on the total weight of the acicular hematite particles or acicular iron oxide hydroxide particles.

25 [0036] In a seventh aspect of the present invention, there are provided a non-magnetic substrate comprising:

a non-magnetic base film; and

a non-magnetic undercoat layer formed on said non-magnetic base film, comprising a binder resin and non-magnetic acicular black iron-based composite particles,

said non-magnetic acicular black iron-based composite particles comprising:

acicular manganese-containing hematite particles or acicular manganese-containing iron oxide hydroxide particles having an average major axis diameter of 0.02 to 0.30 µm;

a coating layer formed on surface of said acicular manganese-containing hematite particle or acicular manganese-containing iron oxide hydroxide particle, comprising at least one organosilicon compound selected from the group consisting of:

- (1) organosilane compounds obtained by drying or heat-treating an alkoxysilane compounds,
- (2) polysiloxanes or modified polysiloxanes, and
- (3) fluoroalkyl organosilane compounds obtained by drying or heat-treating a fluoroalkylsilane compounds; and

carbon black fine particles having a particle size of 0.002 to 0.05  $\mu$ m, adhered on at least a part of said coating layer,

the amount of said carbon black fine particles adhered being 1 to 20 parts by weight based on 100 parts by weight of said acicular manganese-containing hematite particles or acicular manganese-containing iron oxide hydroxide particles.

[0037] In an eighth aspect of the present invention, there are provided a non-magnetic substrate comprising:

a non-magnetic base film; and

a non-magnetic undercoat layer formed on said non-magnetic base film, comprising a binder resin and non-magnetic acicular black iron-based composite particles,

said non-magnetic acicular black iron-based composite particles comprising:

acicular manganese-containing hematite particles or acicular manganese-containing iron oxide hydroxide particles having an average major axis diameter of 0.02 to 0.30 µm;

a coating layer formed on surface of said acicular manganese-containing hematite particle or acicular manganese-containing iron oxide hydroxide particle, comprising at least one organosilicon compound selected from the group consisting of:

- (1) organosilane compounds obtained by drying or heat-treating an alkoxysilane compounds,
- (2) polysiloxanes or modified polysiloxanes, and
- (3) fluoroalkyl organosilane compounds obtained by drying or heat-treating a fluoroalkylsilane compounds; and

carbon black fine particles having a particle size of 0.002 to 0.05 µm, adhered on at least a part of said coating

the amount of said carbon black fine particles adhered being 1 to 20 parts by weight based on 100 parts by weight of said acicular manganese-containing hematite particles or acicular manganese-containing iron oxide hydroxide particles,

said acicular manganese-containing hematite particles or acicular manganese-containing iron oxide hydroxide particles being particles having a coat formed on at least a part of the surface of said acicular manganese-containing hematite particles or acicular manganese-containing iron oxide hydroxide particles and comprising at least one compound selected from the group consisting of hydroxides of aluminum, oxides of aluminum, hydroxides of silicon and oxides of silicon in an amount of 0.01 to 50 % by weight (calculated as Al or SiO<sub>2</sub>) based on the total weight of the acicular manganese-containing hematite particles or acicular manganese-containing iron oxide hydroxide particles.

## [0038] In the accompanying drawings

20

25

5

10

15

- Fig. 1 is an electron photograph (x 60,000) showing a particle structure of acicular manganese-containing hematite particles used in Example 1;
- Fig. 2 is an electron photograph (x 60,000) showing a particle structure of carbon black fine particles used in Example 1;
- Fig. 3 is an electron photograph (× 60,000) showing a particle structure of non-magnetic acicular black iron-based composite particles obtained in Example 1; and
- Fig. 4 is an electron photograph (× 60,000) showing a particle structure of mixed particles of acicular manganesecontaining hematite particles and carbon black fine particles for comparative purposes.
- [0039] A magnetic recording medium and a non-magnetic substrate therefor according to the present invention is 30 described.
  - [0040] The magnetic recording medium according to the present invention comprises:
    - a non-magnetic base film;
    - a non-magnetic undercoat layer formed on the non-magnetic base film, comprising a binder resin and non-magnetic acicular black iron-based composite particles; and a magnetic coating film formed on the non-magnetic undercoat layer, comprising a binder resin and magnetic particles.
  - [0041] The non-magnetic substrate according to the present invention comprises:

40

50

- a non-magnetic base film; and
- a non-magnetic undercoat layer formed on the non-magnetic base film, comprising a binder resin and the nonmagnetic acicular black iron-based composite particles.
- [0042] The non-magnetic particles contained in a non-magnetic undercoat layer according to the present invention are non-magnetic acicular black iron-based composite particles comprising:
  - acicular hematite particles or acicular iron oxide hydroxide particles having an average major axis diameter of 0.02 to 0.30 μm;
  - a coating layer formed on surface of the acicular hematite particle or acicular iron oxide hydroxide particle, comprising at least one organosilicon compound,
    - carbon black fine particles having a particle size of 0.002 to 0.05 µm, adhered on at least a part of the coating layer.
  - The acicular hematite particles assume usually a red color and the acicular iron oxide hydroxide particles assume usually yellow color. In order to produce non-magnetic acicular black iron-based composite particles having a more excellent blackness, it is preferred to use acicular manganese-containing hematite particles or acicular manganese-containing goethite particles, both which contain manganese in an amount of 5 to 40 % by weight based on the weight of the acicular manganese-containing hematite particles or acicular manganese-containing goethite particles,

as the acicular hematite particles or manganese-containing goethite particles used as the core particles of the non-magnetic acicular black iron-based composite particles used in the present invention.

**[0044]** The particle shape of the acicular hematite particles or the acicular iron oxide hydroxide particles as the core particles may include not only acicular shape but also spindle shape, rice ball shape or the like.

[0045] The average major axis diameter of the acicular hematite particles or the acicular iron oxide hydroxide particles as the core particles is usually 0.02 to 0.3  $\mu$ m. If the average major axis diameter thereof exceeds 0.3  $\mu$ m, since the average major axis diameter of the obtained non-magnetic acicular black iron-based composite particles therefrom exceeds 0.3  $\mu$ m, the surface smoothness of the coating film formed using such particles may be impaired. On the other hand, if the average major axis diameter thereof is less than 0.02  $\mu$ m, the dispersion in the vehicle may be difficult because of the increase of the intermolecular force due to the fine particles. With the consideration of the dispersibility in the vehicle and the surface smoothness of the coating film, the average major axis diameter thereof is preferably 0.025 to 0.25  $\mu$ m, more preferably 0.03 to 0.2  $\mu$ m.

[0046] The average minor axis diameter of the acicular hematite particles or the acicular iron oxide hydroxide particles as the core particles is usually 0.01 to 0.15  $\mu$ m. If the average minor axis diameter thereof exceeds 0.15  $\mu$ m, since the average minor axis diameter of the obtained non-magnetic acicular black iron-based composite particles therefrom exceeds 0.15  $\mu$ m, the surface smoothness of the coating film formed using such particles may be impaired. On the other hand, if the average minor axis diameter thereof is less than 0.01  $\mu$ m, the dispersion in the vehicle may be difficult because of the increase of the intermolecular force due to the fine particles. With the consideration of the dispersibility in the vehicle and the surface smoothness of the coating film, the average minor axis diameter thereof is preferably 0.0125 to 0.125  $\mu$ m, more preferably 0.05 to 0.1  $\mu$ m.

[0047] The aspect ratio (= average major axis diameter: average minor axis diameter, hereinafter referred to merely as "aspect ratio") of the acicular hematite particles or the acicular iron oxide hydroxide particles as the core particles is usually 2:1 to 20:1. When the aspect ratio is more than 20:1, the particles may be entangled with each other in vehicle, thereby causing a tendency that the dispersibility thereof may be deteriorated or the viscosity thereof may be increased. On the other hand, when the aspect ratio is less than 2:1, it may be difficult to obtain a coating film having a sufficient strength. With the consideration of the dispersibility in the vehicle and the strength of the coating film, the aspect ratio is preferably 2.5:1 to 18:1, more preferably 3:1 to 15:1.

[0048] The BET specific surface area ( $S_{BET}$ ) of the acicular hematite particles or the acicular iron oxide hydroxide particles as the core particles is usually 35 to 250 m²/g. If the BET specific surface area ( $S_{BET}$ ) thereof is more than 250 m²/g, the dispersion in the vehicle may be difficult because of the increase of the intermolecular force due to the fine particles. On the other hand, if the BET specific surface area ( $S_{BET}$ ) thereof is less than 35 m²/g, the acicular hematite particles or the acicular iron oxide hydroxide particles may be coarse particles or large particles produced by sintering a particle and between particles, which are apt to exert a deleterious influence on the surface smoothness of the coating film. With the consideration of the dispersibility in the vehicle and the surface smoothness of the coating film, the BET specific surface area ( $S_{BET}$ ) thereof is preferably 38 to 200 m²/g, more preferably 40 to 180 m²/g.

[0049] The geometrical standard deviation of the major axis diameter of the acicular hematite particles or the acicular iron oxide hydroxide particles as the core particles is usually not more than 1.50. If the geometrical standard deviation of the major axis diameter thereof exceeds 1.50, the coarse particles existent sometimes exert a deleterious influence on the surface smoothness of the coating film. With the consideration of the surface smoothness of the coating film, the upper limit thereof is preferably 1.45, more preferably not more than 1.40. From the point of view of industrial productivity, the lower limit thereof is preferably 1.01.

[0050] With respect of the blackness of the acicular hematite particles as the core particles, in case of the acicular hematite particles, the lower limit of the blackness thereof, when represented by the L\* value, is usually 18, and the upper limit thereof is usually 28, preferably 25. In the case of the acicular manganese-containing hematite particles, the lower limit of the blackness thereof as the core particles when represented by the L\* value, is usually 18, and the upper limit thereof is usually 27, preferably 24.

**[0051]** With respect to the blackness of the acicular iron oxide hydroxide particles as the core particles, in the case of the acicular goethite particles, the lower limit of the blackness thereof, when represented by the L\* value, is usually 18, and the upper limit thereof is usually 30, preferably 28, and in the case of the acicular manganese-containing goethite particles, the lower limit of the blackness thereof, when represented by the L\* value, is usually 18, and the upper limit thereof is usually 29, preferably 27.

**[0052]** When the L\* value is more than the above upper limit, the blackness of the core particles is insufficient, thereby failing to obtain the non-magnetic acicular black iron-based composite particles having an excellent blackness.

**[0053]** The volume resistivity of the acicular hematite particles or acicular iron oxide hydroxide particles as the core particles usually not more than  $1 \times 10^8 \,\Omega$  • cm.

[0054] The coating layer formed on the surfaces of the core particles comprises at least one organosilicon compound selected from the group consisting of (1) organosilane compounds obtained by drying or heat-treating alkoxysilane compounds; (2) polysiloxanes, or modified polysiloxanes selected from the group consisting of (A) polysiloxanes mod-

ified with at least one compound selected from the group consisting of polyethers, polyesters and epoxy compounds (hereinafter referred to merely as "modified polysiloxanes"), and (B) polysiloxanes whose molecular terminal is modified with at least one group selected from the group consisting of carboxylic acid groups, alcohol groups and a hydroxyl group; and (3) fluoroalkyl organosilane compounds obtained by drying or heat-treating fluoroalkylsilane compounds.

[0055] The organosilane compounds (1) can be produced by drying or heat-treating alkoxysilane compounds represented by the formula (I):

$$R^{1}_{a}SiX_{4-a} \tag{I}$$

wherein R<sup>1</sup> is C<sub>6</sub>H<sub>5</sub>-, (CH<sub>3</sub>)<sub>2</sub>CHCH<sub>2</sub>- or n-C<sub>b</sub>H<sub>2b+1</sub>- (wherein b is an integer of 1 to 18); X is CH<sub>3</sub>0- or C<sub>2</sub>H<sub>5</sub>0-; and a is an integer of 0 to 3. However, R<sup>1</sup> may be any C<sub>1</sub>-C<sub>18</sub> alkyl group, for example C<sub>1</sub>-C<sub>10</sub> alkyl or C<sub>1</sub>-C<sub>4</sub> alkyl. The alkyl group may be a straight-chain alkyl group or a branched alkyl group. It may therefore be a normal (n-) alkyl group.

[0056] The drying or heat-treatment of the alkoxysilane compounds is conducted, for example, at a temperature of usually 40 to 200°C, preferably 60 to 150°C for usually 10 minutes to 12 hours, preferably 30 minutes to 3 hours.

[0057] Specific examples of the alkoxysilane compounds may include methyl triethoxysilane, dimethyl diethoxysilane, tetraethoxysilane, phenyl triethyoxysilane, diphenyl diethoxysilane, methyl trimethoxysilane, dimethyl dimethoxysilane, tetramethoxysilane, phenyl trimethoxysilane, diphenyl dimethoxysilane, isobutyl trimethoxysilane, decyl trimethoxysilane or the like. Among these alkoxysilane compounds, in view of the desorption percentage and the adhering effect of carbon black fine particles, methyl triethoxysilane, phenyl triethyoxysilane, methyl trimethoxysilane, dimethyl dimethoxysilane and isobutyl trimethoxysilane are preferred, and methyl triethoxysilane and methyl trimethoxysilane are more preferred.

[0058] As the polysiloxanes (2), there may be used those compounds represented by the formula (II):

wherein  $R^2$  is H- or  $CH_3$ -, and d is an integer of 15 to 450.

**[0059]** Among these polysiloxanes, in view of the desorption percentage and the adhering effect of carbon black fine particles, polysiloxanes having methyl hydrogen siloxane units are preferred.

[0060] As the modified polysiloxanes (A), there may be used:

(a) polysiloxanes modified with polyethers represented by the formula (III):

wherein  $R^3$  is -(-CH<sub>2</sub>-)<sub>h</sub>-;  $R^4$  is -(-CH<sub>2</sub>-)<sub>i</sub>-CH<sub>3</sub>;  $R^5$  is -OH, - COOH, -CH=CH<sub>2</sub>, -C(CH<sub>3</sub>)=CH<sub>2</sub> or -(-CH<sub>2</sub>-)<sub>j</sub>-CH<sub>3</sub>;  $R^6$  is -(-CH<sub>2</sub>-)<sub>k</sub>-CH<sub>3</sub>; g and h are an integer of 1 to 15; i, j and k are an integer of 0 to 15; e is an integer of 1 to 50; and f is an integer of 1 to 300;

(b) polysiloxanes modified with polyesters represented by the formula (IV):

55

5

15

25

30

35

40

45

wherein  $R^7$ ,  $R^8$  and  $R^9$  are -(-CH<sub>2</sub>-)<sub>q</sub>- and may be the same or different;  $R^{10}$  is -OH, -COOH, -CH=CH<sub>2</sub>, -C(CH<sub>3</sub>)=CH<sub>2</sub> or -(-CH<sub>2</sub>-)<sub>r</sub>-CH<sub>3</sub>;  $R^{11}$  is -(-CH<sub>2</sub>-)<sub>s</sub>-CH<sub>3</sub>; n and q are an integer of 1 to 15; r and s are an integer of 0 to 15; e' is an integer of 1 to 50; and f' is an integer of 1 to 300;

(c) polysiloxanes modified with epoxy compounds represented by the formula (V):

5

10

15

20

25

30

35

40

50

55

wherein  $R^{12}$  is -(-CH<sub>2</sub>-)<sub>v</sub>-; v is an integer of 1 to 15; t is an integer of 1 to 50; and u is an integer of 1 to 300; or a mixture thereof.

[0061] Among these modified polysiloxanes (A), in view of the desorption percentage and the adhering effect of carbon black fine particles, the polysiloxanes modified with the polyethers represented by the formula (III), are preferred.

[0062] As the terminal-modified polysiloxanes (B), there may be used those represented by the formula (VI):

$$R^{13} - \stackrel{\text{CH}_3}{\stackrel{\text{I}}{\stackrel{\text{CH}_3}}{\stackrel{\text{CH}_3}{\stackrel{\text{CH}_3}{\stackrel{\text{CH}_3}{\stackrel{\text{CH}_3}{\stackrel{\text{CH}_3}}{\stackrel{\text{CH}_3}{\stackrel{\text{CH}_3}{\stackrel{\text{CH}_3}{\stackrel{\text{CH}_3}{\stackrel{\text{CH}_3}}{\stackrel{\text{CH}_3}{\stackrel{\text{CH}_3}{\stackrel{\text{CH}_3}}{\stackrel{\text{CH}_3}{\stackrel{\text{CH}_3}}{\stackrel{\text{CH}_3}{\stackrel{\text{CH}_3}}{\stackrel{\text{CH}_3}{\stackrel{\text{CH}_3}}{\stackrel{\text{CH}_3}{\stackrel{\text{CH}_3}}{\stackrel{\text{CH}_3}{\stackrel{\text{CH}_3}}{\stackrel{\text{CH}_3}{\stackrel{\text{CH}_3}}{\stackrel{\text{CH}_3}{\stackrel{\text{CH}_3}}{\stackrel{\text{CH}_3}}{\stackrel{\text{CH}_3}}{\stackrel{\text{CH}_3}}{\stackrel{\text{CH}_3}{\stackrel{\text{CH}_3}}}{\stackrel{\text{CH}_3}}{\stackrel{\text{CH}_3}}{\stackrel{\text{CH}_3}}}{\stackrel{\text{CH}_3}}{\stackrel{\text{CH}_3}}}{\stackrel{\text{CH}_3}}{\stackrel{CH}_3}}{\stackrel{CH}_3}}}}}}}}}}}}}}}}}}}}}}}$$

wherein  $R^{13}$  and  $R^{14}$  are -OH,  $R^{16}$ OH or  $R^{17}$ COOH and may be the same or different;  $R^{15}$  is -CH<sub>3</sub> or -C<sub>6</sub>H<sub>5</sub>;  $R^{16}$  and  $R^{17}$  are -(-CH<sub>2</sub>-)<sub>y</sub>-; y is an integer of 1 to 15; w is an integer of 1 to 200; and x is an integer of 0 to 100.

[0063] Among these terminal-modified polysiloxanes, in view of the desorption percentage and the adhering effect of carbon black fine particles, the polysiloxanes whose terminals are modified with carboxylic acid groups are preferred.

[0064] The fluoroalkyl organosilane compounds (3) may be produced by drying or heat-treating fluoroalkylsilane compounds represented by the formula (VII):

$$CF_3(CF_2)_7CH_2CH_2(R^{18})_{a'}SiX_{4-a'}$$
 (VII)

wherein  $R^{18}$  is  $CH_3$ -,  $C_2H_5$ -,  $CH_3$ O- or  $C_2H_5$ O-; X is  $CH_3$ O- or  $C_2H_5$ O-; and z is an integer of 0 to 15; and a' is an integer of 0 to 3.

**[0065]** The drying or the heat-treatment of the fluoroalkylsilane compounds may be conducted, for example, at a temperature of usually 40 to 200°C, preferably 60 to 150°C for usually 10 minutes to 12 hours, preferably 30 minutes to 3 hours.

[0066] Specific examples of the fluoroalkylsilane compounds may include trifluoropropyl trimethoxysilane, tridecafluorooctyl trimethoxysilane, heptadecafluorodecyl trimethoxysilane, heptadecafluorodecylmethyl dimethoxysilane, trifluoropropyl triethoxysilane, tridecafluorooctyl triethoxysilane, heptadecafluorodecyl triethoxysilane, h

decylmethyl diethoxysilane or the like. Among these fluoroalkylsilane compounds, in view of the desorption percentage and the adhering effect of carbon black fine particles, trifluoropropyl trimethoxysilane, tridecafluorooctyl trimethoxysilane and heptadecafluorodecyl trimethoxysilane are preferred, and trifluoropropyl trimethoxysilane and tridecafluorooctyl trimethoxysilane are more preferred.

[0067] The coating amount of the organosilicon compounds is usually 0 02 to 5.0 % by weight, preferably 0.03 to 2.0 % by weight, more preferably 0.05 to 1.5 % by weight (calculated as Si) based on the weight of the acicular hematite particles or the acicular iron oxide hydroxide particles coated with the organosilicon compounds.

[0068] When the coating amount of the organosilicon compounds is less than 0.02 % by weight, it may become difficult to adhere carbon black fine particles on the coating layer in such an amount enough to improve the blackness and volume resistivity thereof. On the other hand, even when the coating amount of the organosilicon compounds is more than 5.0 % by weight, a sufficient amount of carbon black fine particles can be adhered on the coating layer. However, it is meaningless because the blackness cannot be further improved by using such an excess amount of the organosilicon compounds.

[0069] As the carbon black fine particles used in the non-magnetic acicular black iron-based composite particles in the present invention, there may be exemplified commercially available carbon blacks such as furnace black or channel black. Specific examples of the commercially available carbon blacks usable in the present invention, may include #3050, #3150, #3250, #3750, #3950, MA-100, MA7, #1000, #2400B, #30, MA77, MA8, #650, MA11, #50, #52, #45, #2200B, MA600, etc. (tradename, produced by MITSUBISHI CHEMICAL CORP.), SEAST 9H, SEAST 7H, SEAST 6, SEAST 3H, SEAST 300, SEAST FM, etc. (tradename, produced by TOKAI CARBON CO., LTD.) or the like. In view of the compatibility with the organosilicon compounds, #3150, #3250, MA-100, MA7, #1000, #2400B and #30 are preferred. Further, with the consideration of the conductivity, #3150 and #3250 are more preferred.

[0070] The average major axis diameter of the carbon black fine particles is usually 0.002 to  $0.05~\mu m$ , preferably 0.002 to  $0.035~\mu m$ . When the average major axis diameter of the carbon black fine particles is less than  $0.002~\mu m$ , the carbon black fine particles are too fine to be well handled. On the other hand, when the average major axis diameter is more than  $0.05~\mu m$ , the particle size of the carbon black fine particles may become much larger than that of the acicular hematite particles or the acicular iron oxide hydroxide particles as the core particles, thereby causing insufficient adhesion of the carbon black fine particles onto the coating layer composed of the organosilicon compounds and increasing the desorption percentage of the carbon black fine particles. As a result, the dispersibility of the obtained composite particles in a vehicle is deteriorated.

[0071] The ratio value of an average major axis diameter of the acicular hematite particles or the acicular iron oxide hydroxide particles to an average major axis diameter of the carbon black fine particles is preferably not less than 2. When the ratio value is less than 2, the particle size of the carbon black fine particles may become considerably large as compared to that of the acicular hematite particles or the acicular iron oxide hydroxide particles as the core particles, thereby causing insufficient adhesion of the carbon black fine particles onto the coating layer composed of the organosilicon compounds and increasing the desorption percentage of the carbon black fine particles. As a result, the dispersibility of the obtained composite particles in a vehicle is deteriorated. The upper limit of the ratio value thereof is preferably 150.

35

[0072] The amount of the carbon black fine particles adhered is 1 to 20 parts by weight based on 100 parts by weight of the acicular hematite particles or the acicular iron oxide hydroxide particles as the core particles. When the amount of the carbon black fine particles adhered is less than 1 part by weight, the blackness and volume resistivity of the resultant composite particles is unsatisfactory because of insufficient amount of the carbon black fine particles adhered onto the coating layer. On the other hand, when the amount of the carbon black fine particles adhered is more than 20 parts by weight, the carbon black fine particles tend to be desorbed from the coating layer because of too much amount of the carbon black fine particles adhered thereonto, though the obtained composite particles can show a sufficient blackness and volume resistivity. As a result, the dispersibility of the obtained composite particles in a vehicle is deteriorated.

**[0073]** The particle shape and particle size of the non-magnetic acicular black iron-based composite particles used as non-magnetic particles in the present invention are considerably varied depending upon those of acicular hematite particles or acicular iron oxide hydroxide particles as core particles. Specifically, the non-magnetic acicular black iron-based composite particles used as non-magnetic particles in the present invention are substantially similar in particle shape to that of the core particles, and have a slightly larger particle size than that of the core particles.

[0074] The non-magnetic acicular black iron-based composite particles used in the present invention have an average major axis diameter of usually 0.021 to 0.35  $\mu$ m, preferably 0.026 to 0.30  $\mu$ m, more preferably 0.035 to 0.25  $\mu$ m; an average minor axis diameter of usually 0.0105 to 0.175  $\mu$ m, preferably 0.013 to 0.15  $\mu$ m, more preferably 0.0175 to 0.125  $\mu$ m; an aspect ratio (average major axis diameter/average minor axis diameter) of usually 2:1 to 20:1, preferably 2.5:1 to 18:1, more preferably 3:1 to 15:1; and a BET specific surface area of usually 35 to 250 m²/g, preferably 38 to 200 m²/g, more preferably 40 to 180 m²/g.

[0075] When the average major axis diameter of the non-magnetic acicular black iron-based composite particles is

less than  $0.021~\mu m$ , the intermolecular force between the particles may be increased due to the fineness thereof, so that it may become difficult to uniformly disperse the particles in a vehicle. On the other hand, when the average major axis diameter thereof is more than  $0.35~\mu m$ , since the non-magnetic acicular black iron-based composite particles are coarse, the surface smoothness of the coating film formed using such particles may be impaired.

[0076] Further, it is preferred that the non-magnetic acicular black iron-based composite particles used in the present invention have a geometrical standard deviation of major axis diameter of not more than 1.50. When the geometrical standard deviation of major axis diameter is more than 1.50, since coarse particles tend to be present in the non-magnetic acicular black iron-based composite particles, the surface smoothness of the coating film formed using such particles may be impaired. With the consideration of the surface smoothness, the geometrical standard deviation of major axis diameter of the non-magnetic acicular black iron-based composite particles used in the present invention is preferably not more than 1.45, more preferably not more than 1.40. Further, in view of industrial production of the non-magnetic acicular black iron-based composite particles, the lower limit of the geometrical standard deviation of major axis diameter is preferably 1.01.

[0077] The upper limit of the blackness of the non-magnetic acicular black iron-based composite particles used in the present invention is usually 23.5 when represented by a L\* value thereof. When the L\* value as the upper limit of the blackness is more than 23.5, since the lightness of the non-magnetic acicular black iron-based composite particles is increased, it is difficult to reduce the light transmittance of the magnetic recording medium. The preferable upper limit of the blackness thereof is 23.0. The upper limit of the blackness of the non-magnetic acicular black iron-based composite particles obtained by using the acicular manganese-containing hematite particles or the acicular manganese-containing iron oxide hydroxide particles as the core particles, is usually 22.0, preferably 21.0 when represented by a L\* value.

[0078] The volume resistivity of the non-magnetic acicular black iron-based composite particles used in the present invention is preferably not more than  $1 \times 10^8 \ \Omega \cdot \text{cm}$ , more preferably  $1 \times 10^5 \text{ to } 5 \times 10^7 \ \Omega \cdot \text{cm}$ , still more preferably  $1 \times 10^5 \text{ to } 1 \times 10^7 \ \Omega \cdot \text{cm}$ . When the volume resistivity is more than  $1 \times 10^8 \ \Omega \cdot \text{cm}$ , it is difficult to sufficiently lower the surface resistivity of the magnetic recording medium.

[0079] The percentage of desorption of carbon black fine particles from the non-magnetic acicular black iron-based composite particles used in the present invention is usually not more than 20 %, preferably not more than 10 %. When the desorption percentage of carbon black fine particles is more than 20 %, the desorbed carbon black fine particles tend to inhibit the composite particles from being uniformly dispersed in a vehicle upon the production of non-magnetic coating composition.

[0080] In the non-magnetic acicular black iron-based composite particles used in the present invention, the surfaces of the acicular hematite particles or the acicular iron oxide hydroxide particles as the core particles may be preliminarily coated with at least one compound selected from the group consisting of hydroxide of aluminum, oxides of aluminum, hydroxides of silicon and oxides of silicon (hereinafter referred to as "undercoat composed of hydroxides or oxides of aluminum or silicon"), if required. In this case, the dispersibility of the obtained composite particles in a vehicle may become improved as compared to those having no undercoat composed of hydroxides or oxides of aluminum or silicon. [0081] The coating amount of the hydroxides or oxides of aluminum or silicon is 0.01 to 50 % by weight calculated as Al, SiO<sub>2</sub> or a sum of Al and SiO<sub>2</sub>, based on the weight of the acicular hematite particles or the acicular iron oxide hydroxide particles as the core particles. When the coating amount of the hydroxides or oxides of aluminum or silicon is less than 0.01 % by weight, the improvement of the dispersibility of the obtained composite particles in a vehicle cannot be achieved. On the other hand, when the coating amount of the hydroxides or oxides of aluminum or silicon is more than 50 % by weight, the obtained composite particles can exhibit a good dispersibility in a vehicle, but it is meaningless because the dispersibility cannot be further improved by using such an excess coating amount of the hydroxides or oxides of aluminum or silicon.

[0082] The non-magnetic acicular black iron-based composite particles having the undercoat composed of the hydroxides or oxides of aluminum or silicon may be substantially identical in a particle size, a geometrical standard deviation, a BET specific surface area, a blackness (L\* value) and a volume resistivity, to those having no undercoat composed of the hydroxides or oxides of aluminum or silicon.

**[0083]** Next, the process for producing the non-magnetic acicular black iron-based composite particles used in the present invention, is described.

[0084] The acicular goethite particles as the core particles may be produced by a so-called wet process, i.e., by passing an oxygen-containing gas such as air through a suspension containing either ferrous hydroxide colloid, iron carbonate or iron-containing precipitates obtained by reacting a ferrous salt aqueous solution with alkali hydroxide, alkali carbonate or both of alkali hydroxide and alkali carbonate, and then after filtering-out and washing with water.

[0085] The acicular hematite particles as the core particles may be produced by heat-treating the obtained acicular goethite particles at a temperature of 250 to 850°C in an oxygen-containing gas such as air.

[0086] The acicular manganese-containing hematite particles as the core particles may be produced by heat-treating acicular goethite particles containing manganese in an amount of 8 to 150 atomic % based on whole Fe which are

obtained by the below-mentioned method, at a temperature of 250 to 850°C in an oxygen-containing gas such as air.

**[0087]** The acicular manganese-containing goethite particles as the core particles may be produced by conducting the above-mentioned wet process for producing the acicular goethite particles, in the presence of manganese, thereby forming acicular goethite particles containing manganese in an amount of 8 to 150 atomic % based on whole Fe.

[0088] Alternatively, elements other than Fe such as Ni, Zn, P and Si, which are generally added in order to enhance various properties of the particles such as the major axis diameter, the minor axis diameter and the aspect ratio, may be added during the reaction system for producing the goethite particles.

**[0089]** Especially, prior to the heat-dehydration of the acicular goethite particles for producing the acicular hematite particles, it is preferred that the surfaces of the acicular goethite particles are coated with sintering preventive, as is well known in the art. The coating treatment of the sintering preventive is composed of the steps of: adding the sintering preventive to an aqueous suspension containing the acicular goethite particles, mixing and stirring the resultant suspension, filtering out the particles, washing the particles with water, and drying the particles.

[0090] As the sintering preventive, known sintering preventives are usable. For example, phosphorus compounds such as sodium hexametaphosphate, polyphospholic acid and orthophosphoric acid, silicon compounds such as #3 water glass, sodium orthosilicate, sodium metasilicate and colloidal silica, boron compounds such as boric acid, aluminum compounds including aluminum salts such as aluminum acetate, aluminum sulfate, aluminum chloride and aluminum nitrate, alkali aluminate such as sodium aluminate, alumina sol and aluminum hydroxide, and titanium compounds such as titanyl sulfate may be exemplified. orthophosphoric acid, colloidal silica, boric acid and aluminum acetate are preferable.

[0091] The coating of the acicular hematite particles or the acicular iron oxide hydroxide particles with the alkoxysilane compounds, the polysiloxanes, the modified polysiloxanes, the terminal-modified polysiloxanes or the fluoroalkylsilane compounds, may be conducted by mechanically mixing and stirring the acicular hematite particles or the acicular iron oxide hydroxide particles together with the alkoxysilane compounds, the polysiloxanes, the modified polysiloxanes or the fluoroalkylsilane compounds; or by mechanically mixing and stirring both the components together while spraying the alkoxysilane compounds, the polysiloxanes, the modified polysiloxanes or the fluoroalkylsilane compounds onto the acicular hematite particles or the acicular iron oxide hydroxide particles. In these cases, substantially whole amount of the alkoxysilane compounds, the polysiloxanes, the modified polysiloxanes, the terminal-modified polysiloxanes or the fluoroalkylsilane compounds added can be applied onto the surfaces of the acicular hematite particles or the acicular iron oxide hydroxide particles.

[0092] In order to uniformly coat the surfaces of the acicular hematite particles or the acicular iron oxide hydroxide particles with the alkoxysilane compounds, the polysiloxanes, the modified polysiloxanes, the terminal-modified polysiloxanes or the fluoroalkylsilane compounds, it is preferred that the acicular hematite particles or the acicular iron oxide hydroxide particles are preliminarily diaggregated by using a pulverizer. As the apparatuses for the mixing and stirring, there may be used an edge runner, a Henschel mixer or the like.

[0093] The conditions for the mixing and stirring such as mixing ratio, linear load, stirring speed or mixing and stirring time, may be appropriately adjusted so as to coat the surfaces of the acicular hematite particles or the acicular iron oxide hydroxide particles with the alkoxysilane compounds, the polysiloxanes, the modified polysiloxanes, the terminal-modified polysiloxanes or the fluoroalkylsilane compounds as uniformly as possible. The mixing and stirring time for the coating treatment is, for example, preferably not less than 20 minutes.

[0094] The amount of the alkoxysilane compounds, the polysiloxanes, the modified polysiloxanes, the terminal-modified polysiloxanes or the fluoroalkylsilane compounds added, is preferably 0.15 to 45 parts by weight based on 100 parts by weight of the acicular hematite particles or the acicular iron oxide hydroxide particles. When the amount of the organosilicon compounds added is less than 0.15 part by weight, it may become difficult to adhere the carbon black fine particles in such an amount enough to improve the blackness and volume resistivity of the obtained composite particles. On the other hand, when the amount of the organosilicon compounds added is more than 45 parts by weight, a sufficient amount of the carbon black fine particles can be adhered on the surface of the coating layer, but it is meaningless because the blackness and volume resistivity of the composite particles cannot be further improved by using such an excess amount of the organosilicon compounds.

**[0095]** Next, the carbon black fine particles are added to the acicular hematite particles or the acicular iron oxide hydroxide particles coated with the organosilicon compounds, and the resultant mixture is mixed and stirred to adhere the carbon black fine particles on the surfaces of the coating layer composed of the organosilicon compounds, and then dried.

[0096] In the case where the alkoxysilane compounds (1) and the fluoroalkylsilane compounds (3) are used as the coating compound, after the carbon black fine particles are adhered on the surface of the coating layer, the resultant composite particles are dried or heat-treated, for example, at a temperature of usually 40 to 200°C, preferably 60 to 150°C for usually 10 minutes to 12 hours, preferably 30 minutes to 3 hours, thereby forming a coating layer composed of the organosilane compounds (1) and the fluoroalkyl organosilane compounds (3), respectively.

[0097] It is preferred that the carbon black fine particles are added little by little and slowly, especially about 5 to 60

minutes.

[0098] The conditions for mixing and stirring the acicular hematite particles or the acicular iron oxide hydroxide particles and the carbon black fine particles coated with the organosilicon compounds, such as mixing ratio, linear load stirring speed or mixing and stirring time, may be appropriately adjusted so as to uniformly adhere the carbon black fine particles on the surface of the coating layer. The mixing and stirring time for the adhesion treatment is, for example, preferably not less than 15 minutes.

[0099] The amount of the carbon black fine particles added, is preferably 1 to 20 parts by weight based on 100 parts by weight of the acicular hematite particles or the acicular iron oxide hydroxide particles. When the amount of the carbon black fine particles added is less than 1 part by weight, it may become difficult to adhere the carbon black fine particles in such an amount enough to improve the blackness and volume resistivity of the obtained composite particles. On the other hand, when the amount of the carbon black fine particles added is more than 20 parts by weight, a sufficient blackness and volume resistivity of the resultant composite particles can be obtained, but the carbon black fine particles tend to be desorbed from the surface of the coating layer because of too large amount of the carbon black fine particles adhered, resulting in deteriorated dispersibility in a vehicle.

**[0100]** The acicular hematite particles or the acicular iron oxide hydroxide particles may be coated with at least one compound selected from the group consisting of hydroxides of aluminum, oxides of aluminum, hydroxides of silicon and oxides of silicon, if required, prior to mixing and stirring with the alkoxysilane compounds, the polysiloxanes, the modified polysiloxanes, the terminal-modified polysiloxanes or the fluoroalkylsilane compounds.

[0101] The coating of the hydroxides or oxides of aluminum or silicon may be conducted by adding an aluminum compound, a silicon compound or both the compounds to a water suspension in which the acicular hematite particles or the acicular iron oxide hydroxide particles are dispersed, followed by mixing and stirring, and further adjusting the pH of the suspension, if required, thereby coating the surfaces of the acicular hematite particles or the acicular iron oxide hydroxide particles with at least one compound selected from the group consisting of hydroxides of aluminum, oxides of aluminum, hydroxides of silicon and oxides of silicon. The thus obtained particles coated with the hydroxides or oxides of aluminum or silicon are then filtered out, washed with water, dried and pulverized. Further, the particles coated with the hydroxides or oxides of aluminum or silicon may be subjected to post-treatments such as deaeration treatment and compaction treatment, if required.

**[0102]** As the aluminum compounds, there may be exemplified aluminum salts such as aluminum acetate, aluminum sulfate, aluminum chloride or aluminum nitrate, alkali aluminates such as sodium aluminate, alumina sols or the like.

[0103] The amount of the aluminum compound added is 0.01 to 50 % by weight (calculated as Al) based on the weight of the acicular hematite particles or the acicular iron oxide hydroxide particles. When the amount of the aluminum compound added is less than 0.01 % by weight, it may be difficult to sufficiently coat the surfaces of the acicular hematite particles or the acicular iron oxide hydroxide particles with hydroxides or oxides of aluminum or silicon, thereby failing to achieve the improvement of the dispersibility in a vehicle. On the other hand, when the amount of the aluminum compound added is more than 50 % by weight, the coating effect is saturated and, therefore, it is meaningless to add such an excess amount of the aluminum compound.

**[0104]** As the silicon compounds, there may be exemplified #3 waterglass, sodium orthosilicate, sodium metasilicate, colloidal silica or the like.

[0105] The amount of the silicon compound added is 0.01 to 50 % by weight (calculated as SiO<sub>2</sub>) based on the weight of the acicular hematite particles or the acicular iron oxide hydroxide particles. When the amount of the silicon compound added is less than 0.01 % by weight, it may be difficult to sufficiently coat the surfaces of the acicular hematite particles or the acicular iron oxide hydroxide particles with hydroxides or oxides of silicon, thereby failing to achieve the improvement of the dispersibility in a vehicle. On the other hand, when the amount of the silicon compound added is more than 50 % by weight, the coating effect is saturated and, therefore, it is meaningless to add such an excess amount of the silicon compound.

**[0106]** In the case where both the aluminum and silicon compounds are used in combination for the coating, the total amount of the aluminum and silicon compounds added is preferably 0.01 to 50 % by weight (calculated as a sum of Al and SiO<sub>2</sub>) based on the weight of the acicular hematite particles or the acicular iron oxide hydroxide particles.

**[0107]** The non-magnetic substrate according to the present invention is produced by forming a coating film on the non-magnetic base film and drying the coating film. The coating film is formed by applying a non-magnetic coating composition which contains the non-magnetic acicular black iron-based composite particles, a binder resin and a solvent, to the surface of the non-magnetic base film.

[0108] As the non-magnetic base film, the following materials which are at present generally used for the production of a magnetic recording medium are usable as a raw material: a synthetic resin such as polyethylene terephthalate, polyethylene, polypropylene, polycarbonate, polyethylene naphthalate, polyamide, polyamide and polyimide; foil and plate of a metal such as aluminum and stainless steel; and various kinds of paper. The thickness of the non-magnetic base film varies depending upon the material, but it is usually about 1.0 to 300  $\mu$ m, preferably 2.0 to 200  $\mu$ m. In the case of a magnetic disc, polyethylene terephthalate is usually used as the non-magnetic base film. The thickness thereof is

usually 50 to 300  $\mu$ m, preferably 60 to 200  $\mu$ m. In the case of a magnetic tape, when polyethylene terephthalate is used as the base film, the thickness thereof is usually 3 to 100  $\mu$ m, preferably 4 to 20  $\mu$ m. When polyethylene naphthalate is used, the thickness thereof is usually 3 to 50  $\mu$ m, preferably 4 to 20  $\mu$ m. When polyamide is used, the thickness thereof is usually 2 to 10  $\mu$ m, preferably 3 to 7  $\mu$ m.

[0109] As the binder resin used in the present invention, the following resins which are at present generally used for the production of a magnetic recording medium are usable: vinyl chloride-vinyl acetate copolymer, urethane resin, vinyl chloride-vinyl acetate-maleic acid copolymer, urethane elastomer, butadiene-acrylonitrile copolymer, polyvinyl butyral, cellulose derivative such as nitrocellulose, polyester resin, synthetic rubber resin such as polybutadiene, epoxy resin, polyamide resin, polyisocyanate, electron radiation curing acryl urethane resin and mixtures thereof. Each of these resin binders may contain a functional group such as -OH, -COOH, -SO<sub>3</sub>M, -OPO<sub>2</sub>M<sub>2</sub> and -NH<sub>2</sub>, wherein M represents H, Na or K. With the consideration of the dispersibility of the particles, a binder resin containing a functional group -COOH or -SO<sub>3</sub>M is preferable.

[0110] The mixing ratio of the non-magnetic acicular black iron-based composite particles with the binder resin is usually 5 to 2000 parts by weight, preferably 100 to 1000 parts by weight based on 100 parts by weight of the binder resin.

[0111] As the solvents, there may be used methyl ethyl ketone, toluene, cyclohexanone, methyl isobutyl ketone, tetrahydrofuran, a mixture of these solvents or the like.

[0112] The total amount of the solvent used is 50 to 1,000 parts by weight based on 100 parts by weight of the non-magnetic acicular black iron-based composite particles. When the amount of the solvent used is less than 50 parts by weight, the viscosity of the non-magnetic coating composition prepared therefrom becomes too high, thereby making it difficult to apply the non-magnetic coating composition. On the other hand, when the amount of the solvent used is more than 1,000 parts by weight, the amount of the solvent volatilized during the formation of the coating film becomes too large, thereby rendering the coating process industrially disadvantageous.

**[0113]** It is possible to add a lubricant, a polishing agent, an antistatic agent, etc. which are generally used for the production of a magnetic recording medium to the non-magnetic undercoat layer.

[0114] The thickness of the non-magnetic undercoat layer obtained by applying a non-magnetic coating composition on the surface of the non-magnetic base film and drying, is usually 0.2 to 10.0  $\mu$ m, preferably 0.5 to 5.0  $\mu$ m. If the thickness is less than 0.2  $\mu$ m, not only it is impossible to ameliorate the surface roughness of the non-magnetic substrate but also the strength is insufficient. If the thickness is more than 10  $\mu$ m, it is difficult to reduce the thickness of the magnetic recording medium.

[0115] The non-magnetic substrate according to the present invention has the following properties.

[0116] In case of using the non-magnetic acicular black iron-based composite particles as non-magnetic particles, in which the organosilicon compound is formed on the surface of the acicular hematite particle or acicular iron oxide hydroxide particle, the non-magnetic substrate according to the present invention has a gloss (of the coating film) of usually 170 to 280%, preferably 180 to 280%, more preferably 185 to 280%; a surface roughness Ra (of the coating film) of usually 0.5 to 15.0 nm, preferably 0.5 to 13.0 nm, more preferably 0.5 to 12.0 nm; a Young's modulus (relative value to a commercially available video tape: and AV T-120 produced by Victor Company of Japan, Limited) of usually 120 to 150, preferably 122 to 150; a linear adsorption coefficient (of the coating film) of usually 1.30 to 5.00  $\mu$ m<sup>-1</sup>; and a surface resistivity of not more than 1 × 10<sup>12</sup>  $\Omega$ /sq, preferably 1 × 10<sup>5</sup> to 3 × 10<sup>11</sup>  $\Omega$ /sq. [0117] In case of using the non-magnetic acicular black iron-based composite particles as non-magnetic particles, in

[0117] In case of using the non-magnetic acicular black iron-based composite particles as non-magnetic particles, in which the organosilicon compound is formed on the surface of the coat composed of the hydroxides and/or oxides of aluminum and/or silicon which are formed on the surface of the acicular hematite particle or acicular iron oxide hydroxide particle, the non-magnetic substrate according to the present invention has a gloss (of the coating film) of usually 175 to 300%, preferably 180 to 300%, more preferably 185 to 300%; a surface roughness Ra (of the coating film) of usually 0.5 to 13.0 nm, preferably 0.5 to 12.0 nm, more preferably 0.5 to 11.0 nm; a Young's modulus (relative value to a commercially available video tape: and AV T-120 produced by Victor Company of Japan, Limited) of usually 122 to 150, preferably 124 to 150; a linear adsorption coefficient (of the coating film) of usually 1.30 to 5.00  $\mu$ m<sup>-1</sup>, preferably 1.35 to 5.00  $\mu$ m<sup>-1</sup>; and a surface resistivity of not more than 1 × 10<sup>12</sup>  $\Omega$ /sq, preferably 1 × 10<sup>5</sup> to 5 × 10<sup>11</sup>  $\Omega$ /sq.

**[0118]** The magnetic recording medium according to the present invention can be produced by applying a magnetic coating composition containing the magnetic particles, a binder resin and a solvent, on the non-magnetic undercoat layer, followed by drying, to form a magnetic recording layer thereon.

[0119] As the magnetic particles used in the present invention, magnetic particles containing iron as a main component are usable, and there may be exemplified magnetic iron oxide particles such as maghemite particles, magnetite particles and berthollide compound particles which are an intermediate oxide between maghemite and magnetite; particles obtained by incorporating any one or more different kinds of elements other than Fe, such as Co, Al, Ni, P, Zn, Si, B or the like in the said magnetic iron oxide particles; Co modified particles obtained by modifying the said magnetic iron oxide particles with cobalt; magnetic acicular metal particles containing iron as a main component and elements other than Fe at least one selected from the group consisting of Co, Al, Ni, P, Si, Zn, B, Nd, La and Y, including magnetic acicular iron-based alloy particles; magnetoplumbite-type ferrite particles such as plate-like ferrite particles containing

Ba, Sr or Ba-Sr; plate-like magnetoplumbite-type ferrite particles obtained by incorporating divalent metals (such as Co, Ni, Zn, Mg, Mn or the like) or tetravalent metals (such as Ti, Sn, Zr or the like) as a coercive force-reducing agent in the plate-like magnetoplumbite-type ferrite particles; or the like. With the consideration of the short-wavelength recording and the high-density recording, magnetic acicular metal particles containing iron as a main component and magnetic acicular iron-based alloy particles containing elements other than Fe at least one selected from the group consisting of Co, Al, Ni, P, Si, Zn, B, Nd, La, Y or the like are preferable.

[0120] The magnetic acicular metal particles containing iron as a main component comprising (i) iron and Al; (ii) iron, Co and Al, (iii) iron, Al and at least one rare-earth metal such as Nd, La and Y, or (iv) iron, Co, Al and at least one rare-earth metal such as Nd, La and Y is even more preferable from the point of the durability of the magnetic recording medium. Further, the magnetic acicular metal particles containing iron as a main component comprising iron, Al and at least one rare-earth metal such as Nd, La and Y is most preferable.

**[0121]** The iron content in the particles is the balance, and is preferably 50 to 99 % by weight, more preferably 60 to 95 % by weight (calculated as Fe) based on the weight of the magnetic acicular metal particles containing iron as a main component.

[0122] The magnetic particles containing iron as a main component used in the present invention have an average major axis diameter of usually 0.01 to 0.50  $\mu$ m, preferably 0.03 to 0.30  $\mu$ m, an average minor axis diameter of usually 0.0007 to 0.17  $\mu$ m, preferably 0.003 to 0.10  $\mu$ m. It is preferred that the shape of the magnetic particles containing iron as a main component is acicular, cubic or plate-like. The acicular shape may include not only needle-shape but also spindle-shape, rice ball-shape, or the like.

[0123] In the case that the shape of the magnetic particles is acicular, the magnetic particles have an aspect ratio of usually not less than 3:1, preferably and not less than 5:1. The upper limit of the aspect ratio is usually 15:1, preferably 10:1 with the consideration of the dispersibility in the vehicle.

[0124] In the case that the shape of the magnetic particles is plate-like, the magnetic particles have an aspect ratio (an average plate surface diameter/average plate thickness) of usually not less than 2:1, preferably and not less than 3:1. The upper limit of the aspect ratio is usually 20:1, preferably 15:1 with the consideration of the dispersibility in the vehicle.

**[0125]** The geometrical standard deviation of the major axis diameter of the magnetic particles used in the present invention is preferably not more than 2.50 in. If it exceeds 2.50, the coarse particles existent sometimes exert a deleterious influence on the surface smoothness of the magnetic recording layer. From the point of view of industrial productivity, the lower limit of the geometrical standard deviation of the major axis diameter is preferably 1.01.

**[0126]** As to the magnetic properties of the magnetic particles used in the present invention, the coercive force is usually 250 to 4000 Oe, and the saturation magnetization is usually 40 to 170 emu/g.

**[0127]** As to the magnetic properties of the magnetic iron oxide particles used in the present invention, the coercive force is usually 250 to 1700 Oe, preferably 300 to 1700 Oe, and the saturation magnetization is usually 60 to 90 emu/g, preferably 65 to 90 emu/g.

**[0128]** As to the magnetic properties of the magnetic acicular metal particles containing iron as a main component used in the present invention, the coercive force is usually 800 to 3500 Oe, preferably 900 to 3500 Oe, and the saturation magnetization is usually 90 to 170 emu/g, preferably 100 to 170 emu/g.

[0129] As to the magnetic properties of the magnetoplumbite-type ferrite particles used in the present invention, the coercive force is usually 800 to 4000 Oe, preferably 900 to 4000 Oe, and the saturation magnetization is usually 40 to 70 emu/g, preferably 45 to 70 emu/g.

**[0130]** As the binder resin for the magnetic recording layer, the same binder resin as that used for the production of the non-magnetic undercoat layer is usable.

[0131] The mixing ratio of the magnetic acicular metal particles containing iron as a main component with the binder resin in the magnetic recording layer is usually 200 to 2000 parts by weight, preferably 300 to 1500 parts by weight based on 100 parts by weight of the binder resin.

**[0132]** As the solvents, there may be used methyl ethyl ketone, toluene, cyclohexanone, methyl isobutyl ketone, tetrahydrofuran, a mixture of these solvents or the like.

[0133] The total amount of the solvent used is 65 to 1,000 parts by weight based on 100 parts by weight of the magnetic particles. When the amount of the solvent used is less than 65 parts by weight, the viscosity of the magnetic coating composition prepared therefrom becomes too high, thereby making it difficult to apply the magnetic coating composition. On the other hand, when the amount of the solvent used is more than 1,000 parts by weight, the amount of the solvent volatilized during the formation of the coating film becomes too large, thereby rendering the coating process industrially disadvantageous.

[0134] It is possible to add a lubricant, a polishing agent, an antistatic agent, etc., which are generally used for the production of a magnetic recording medium to the magnetic recording layer.

**[0135]** The thickness of the magnetic recording layer obtained by applying the magnetic coating composition on the surface of the non-magnetic undercoat layer and dried, is usually in the range of 0.01 to 5.0  $\mu$ m. If the thickness is less

than 0.01  $\mu$ m, uniform coating may be difficult, so that unfavorable phenomenon such as unevenness on the coating surface is observed. On the other hand, when the thickness exceeds 5.0  $\mu$ m, it may be difficult to obtain desired signal recording property due to an influence of diamagnetism. The preferable thickness is in the range of 0.05 to 1.0  $\mu$ m.

[0136] The magnetic recording medium according to the present invention has the following properties.

[0137] The magnetic recording medium according to the present invention has a coercive force of usually 250 to 4000 Oe; a squareness (residual magnetic flux density Br/saturation magnetic flux density Bm) of usually 0.85 to 0.95; a gloss (of the coating film) of usually 130 to 300%; a surface roughness Ra (of the coating film) of usually not more than 12.0 nm; a Young's modulus (relative value to a commercially available video tape: AV T-120 produced by Victor Company of Japan, Limited) of usually not less than 124; a linear adsorption coefficient (of the coating film) of usually 1.20 to 5.00  $\mu$ m<sup>-1</sup>; and a surface resistivity of not more than 1 × 10<sup>10</sup>  $\Omega$ /sq.

[0138] In case of using non-magnetic substrate wherein the non-magnetic acicular black iron-based composite particles in which the organosilicon compound is formed on the surface of the coat composed of the hydroxides and/or oxides of aluminum and/or silicon which are formed on the surface of the acicular hematite particle or acicular iron oxide hydroxide particle, are used as non-magnetic particles, the magnetic recording medium according to the present invention has a coercive force of usually 250 to 4000 Oe; a squareness (residual magnetic flux density Br/saturation magnetic flux density Bm) of usually 0.85 to 0.95; a gloss (of the coating film) of usually 135 to 300%; a surface roughness Ra (of the coating film) of usually not more than 11.8 nm; a Young's modulus (relative value to a commercially available video tape: AV T-120 produced by Victor Company of Japan, Limited) of usually not less than 126; a linear adsorption coefficient (of the coating film) of usually 1.20 to 5.00  $\mu$ m<sup>-1</sup>; and a surface resistivity of not more than 1  $\times$  10<sup>10</sup>  $\Omega$ /sq.

[0139] In case of using magnetic iron oxide particles as the magnetic particles, the magnetic properties of the magnetic recording medium according to the present invention are a coercive force of usually 250 to 1700 Oe, preferably 300 to 1700 Oe; a squareness (residual magnetic flux density Br/saturation magnetic flux density Bm) of usually 0.85 to 0.95, preferably 0.86 to 0.95.

**[0140]** In case of using magnetic acicular metal particles containing iron as a main component as the magnetic particles, the magnetic properties of the magnetic recording medium according to the present invention are a coercive force of usually 800 to 3500 Oe, preferably 900 to 3500 Oe; a squareness (residual magnetic flux density Br/saturation magnetic flux density Bm) of usually 0.87 to 0.95, preferably 0.88 to 0.95.

[0141] In case of using plate-like magnetoplumbite-type ferrite particles as the magnetic particles, the magnetic properties of the magnetic recording medium according to the present invention are a coercive force of usually 800 to 4000 Oe, preferably 900 to 4000 Oe; a squareness (residual magnetic flux density Br/saturation magnetic flux density Bm) of usually 0.85 to 0.95, preferably 0.86 to 0.95.

[0142] Especially, in case of using the non-magnetic substrate as set forth the fifth aspect and magnetic acicular metal particles containing iron as a main component as the magnetic particles, the magnetic recording medium according to the present invention has a gloss (of the coating film) of usually 180 to 300%, preferably 190 to 300%; a surface roughness Ra (of the coating film) of usually not more than 11.0 nm, preferably 2.0 to 10.5 nm, more preferably 2.0 to 10.0 nm; a Young's modulus (relative value to a commercially available video tape: AV T-120 produced by Victor Company of Japan, Limited) of usually 124 to 160, preferably 128 to 160; a linear adsorption coefficient (of the coating film) of usually 1.20 to 5.00  $\mu$ m<sup>-1</sup>, preferably 1.25 to 5.00  $\mu$ m<sup>-1</sup>; and a surface resistivity of not more than 1 × 10<sup>10</sup>  $\Omega$ /sq, preferably not more than 7.5 × 10<sup>9</sup>  $\Omega$ /sq.

[0143] In case of using the non-magnetic substrate as set forth the sixth aspect and magnetic acicular metal particles containing iron as a main component as the magnetic particles, the magnetic recording medium according to the present invention has a gloss (of the coating film) of usually 185 to 300%, preferably 195 to 300%; a surface roughness Ra (of the coating film) of usually not more than 10.5 nm, preferably 2.0 to 10.0 nm, more preferably 2.0 to 9.5 nm; a Young's modulus (relative value to a commercially available video tape: AV T-120 produced by Victor Company of Japan, Limited) of usually 126 to 160, preferably 130 to 160; a linear adsorption coefficient (of the coating film) of usually 1.20 to 5.00 μm<sup>-1</sup>, preferably 1.25 to 5.00 μm<sup>-1</sup>; and a surface resistivity of not more than 1 × 10<sup>10</sup> Ω/sq, preferably not more than 7.5 × 10<sup>9</sup> Ω/sq.

[0144] The essential of the present invention lies in such a fact that in the case where the non-magnetic acicular black iron-based composite particles comprising: acicular hematite particles or acicular iron oxide hydroxide particles having an average major axis diameter of 0.02 to 0.3 µm, which may be coated with at least one compound selected from the group consisting of hydroxides of aluminum, oxides of aluminum, hydroxides of silicon and oxides of silicon; a coating layer formed on surfaces of said particles, comprising at least one organosilicon compound selected from the group consisting of (1) organosilane compounds obtained by drying or heat-treating alkoxysilane compounds, (2) polysiloxanes, or modified polysiloxanes and (3) fluoroalkyl organosilane compounds obtained by drying or heat-treating fluoroalkylsilane compounds; and carbon black fine particles having a particle size of 0.002 to 0.05 µm, adhered on the coating layer which are contained in a total amount of 1 to 20 parts by weight based on 100 parts by weight of the acicular hematite particles or acicular iron oxide hydroxide particles, are used as non-magnetic particles for the non-magnetic undercoat layer, since the amount of the carbon black fine particles desorbed from the surface of the non-magnetic undercoat layer, since the amount of the carbon black fine particles desorbed from the surface of the non-

magnetic acicular black iron-based composite particle is small, it is possible to more improve a surface smoothness of the non-magnetic undercoat layer and a strength of the non-magnetic substrate, and to more reduce a light transmittance and electric resistance thereof even when the amount of carbon black contained is reduced as low as possible. Further, in the case where a magnetic recording layer is formed on such a non-magnetic undercoat layer, it is possible to obtain a magnetic recording medium having a more smooth surface, a high strength, and a lower light transmittance and a lower electric resistance even when the amount of carbon black in the magnetic recording layer is reduced as low as possible.

**[0145]** The reason why the amount of the carbon black fine particles desorbed from the surfaces of the non-magnetic acicular black iron-based composite particles is small, is considered such that the organosilicon compounds onto which the carbon black fine particles are adhered, are strongly bonded to the surface of the acicular hematite particle or the acicular iron oxide hydroxide particle as a core particle.

[0146] Especially, in the case of using the alkoxysilane compounds or the fluoroalkylsilane compounds, metallosiloxane bonds (=Si-O-M, wherein M represents a metal atom contained in the acicular hematite particle or the acicular iron oxide hydroxide particle as a core particle, such as Si, Al or Fe) are formed between the metal elements such as Si, Al or Fe which are contained within the acicular hematite particle or the acicular iron oxide hydroxide particle or present at the surface thereof, and alkoxy groups of the alkoxysilane compounds or the fluoroalkylsilane compounds on which the carbon black fine particles are adhered, so that the organosilicon compounds onto which the carbon black fine particles are adhered, can be more strongly bonded to the surface of the acicular hematite particle or the acicular iron oxide hydroxide particle.

[0147] The reason why the black iron-based composite particles used in the present invention can show an excellent dispersibility in vehicles is considered as follows. That is, due to the face that the amount of the carbon black fine particles desorbed from the surface of the non-magnetic acicular black iron-based composite particle is small, it is unlikely that the dispersion in the system is inhibited by the carbon black fine particles desorbed. Further, since the carbon black fine particles are adhered on the surface of the non-magnetic acicular black iron-based composite particle, irregularities are formed thereon, because the contact between the particles is suppressed.

[0148] The reason why the substrate and the magnetic recording medium can show a low light transmittance even at a small carbon black content, is considered such that in the non-magnetic acicular black iron-based composite particles, the carbon black particles which ordinarily act as agglomerates due to fineness thereof, can be uniformly and densely adhered on surfaces of the acicular hematite particles or acicular iron oxide hydroxide particles, and can be dispersed in such a condition close to primary particles, thereby enabling individual carbon black fine particles to effectively exhibit properties thereof.

[0149] The reason why the substrate and the magnetic recording medium can show a lower electric resistance even at a small carbon black content, is considered such that since the acicular hematite particles or the acicular iron oxide hydroxide particles are uniformly dispersed in a coating film while keeping a mutual contact between surfaces thereof, the carbon black fine particles uniformly and densely adhered to the surfaces of the acicular hematite particles or acicular iron oxide hydroxide particles, can also be continuously connected with each other while keeping a mutual contact therebetween.

[0150] In accordance with the present invention, due to the fact that the non-magnetic acicular black iron-based composite particles as non-magnetic particles have an excellent dispersibility, it is possible to improve a surface smoothness of the non-magnetic undercoat layer and a strength of the non-magnetic substrate. In addition, due to the fact that the non-magnetic acicular black iron-based composite particles as non-magnetic particles can show an excellent blackness and a low electric resistance even at a small carbon black content, it is possible to reduce a light transmittance and an electric resistance of the non-magnetic substrate. Thus, by using such a non-magnetic undercoat layer, the obtained magnetic recording medium can also exhibit a more smooth surface, a high strength, and a lower light transmittance and a lower electric resistance even when the amount of carbon black contained therein is reduced as low as possible. Accordingly, the magnetic recording medium according to the present invention can be suitably applied to those for high-density recording.

**[0151]** The non-magnetic acicular black iron-based composite particles used in the present invention have an excellent dispersibility in vehicles, resulting in excellent handling property and workability and, therefore, are industrially advantageous.

**[0152]** Further, the magnetic recording medium according to the present invention has a small carbon black content and, therefore, is favorable in view of safety and hygiene.

### **EXAMPLES**

55

**[0153]** The present invention is described in more detail by Examples and Comparative Examples, but the Examples are only illustrative and, therefore, not intended to limit the scope of the present invention.

[0154] Various properties were evaluated by the following methods.

- (1) The average major axis diameters of acicular hematite particles acicular iron oxide hydroxide particles, carbon black fine particles, non-magnetic acicular black iron-based composite particles and magnetic particles were respectively expressed by average values (measured in a predetermined direction) of about 350 particles which were sampled from a micrograph obtained by magnifying an original electron micrograph (x 20,000) by four times in each of the longitudinal and transverse directions.
- (2) The <u>aspect ratio</u> of the particles was expressed by a ratio of average major axis diameter to minor axis diameter thereof.
- (3) The geometrical standard deviation of particle sizes was expressed by values obtained by the following method. That is, the particle sizes were measured from the above-magnified photograph. The actual particle sizes and the number of the particles were obtained from the calculation on the basis of the measured values. On a logarithmic normal probability paper, the particle sizes were plotted at regular intervals on the abscissa-axis and the accumulative number of particles belonging to each interval of the particle sizes were plotted by percentage on the ordinate-axis by a statistical technique. The particle sizes corresponding to the number of particles of 50 % and 84.13 %, respectively, were read from the graph, and the geometrical standard deviation was measured from the following formula:

#### Geometrical standard deviation =

{particle size corresponding to 84.13 % under integration sieve}/{particle size (geometrical average diameter) corresponding to 50 % under integration sieve}

The more the geometrical standard deviation nears 1.0, the more excellent the particle size distribution of the particles.

(4) The specific surface area was expressed by values measured by a BET method.

5

10

15

20

25

30

35

40

45

50

55

- (5) The <u>amounts of Mn, Al and Si</u> which were present within acicular hematite particles, acicular iron oxide hydroxide particles and non-magnetic acicular black iron-based composite particles or on surfaces thereof, and the <u>amount of Si contained in organosilicon compounds</u>, were measured by a fluorescent X-ray spectroscopy device 3063 (manufactured by RIGAKU DENKI KOGYO CO., LTD.) according to JIS K0119 "General rule of fluorescent X-ray analysis".
- (6) The <u>amount of carbon</u> adhered (attached) on the non-magnetic acicular black iron-based composite particles was measured by "Horiba Metal, Carbon and Sulfur Analyzer EMIA-2200 Model" (manufactured by HORIBA SEI-SAKUSHO CO., LTD.).
- (7) The <u>blackness</u> of acicular hematite particles, acicular iron oxide hydroxide particles and non-magnetic acicular black iron-based composite particles, were measured by the following method. That is, 0.5 g of sample particles and 0.7 cc of castor oil were intimately kneaded together by a Hoover's muller to form a paste. 4.5 g of clear lacquer was added to the obtained paste and was intimately mixed to form a paint. The paint was applied on a cast-coated paper by using a 6-mil applicator to produce a coating film piece (having a film thickness of about 30 μm). The thus obtained coating film piece was measured according to JIS Z 8729 by a multi-light source spectrographic colorimeter MSC-IS-2D (manufactured by SUGA TESTING MACHINES MANUFACTURING CO., LTD.) to determine a L\* value of colorimetric indices thereof.
- (8) The <u>volume resistivity</u> of the acicular hematite particles, the acicular iron oxide hydroxide particles and the non-magnetic acicular black iron-based composite particles were measured as follows. First, 0.5 g of the respective particles were weighed, and pressure-molded into a cylindrical shape at 140 Kg/cm<sup>2</sup> using a KBr tablet machine (manufactured by SIMAZU SEISAKUSHO CO., LTD.), thereby producing a cylindrical sample to be measured.

The thus-produced sample was then exposed to an atmosphere kept at a temperature of 25°C and a relative humidity of 60%, for not less than 12 hours. Thereafter, the sample was fixed between stainless steel electrodes, and a voltage of 15V was applied to the sample using a Wheatstone bridge (TYPE2768, manufactured by YOKOGAWA-HOKUSHIN DENKI CO., LTD.), thereby measuring a resistance value R ( $\Omega$ ) of the sample.

Next, an upper surface area A (cm<sup>2</sup>) and a thickness  $t_0$  (cm) of the cylindrical sample were measured, and the respective measured values were substituted for A and  $t_0$  of the following formula to obtain the volume resistivity X ( $\Omega \cdot$  cm) of the sample.

$$X (\Omega \cdot cm) = R \times (A/t_0)$$

5

10

15

25

30

35

40

45

50

55

(9) The <u>desorption percentage (T %)</u> of carbon black fine particles adhered on the non-magnetic acicular black iron-based composite particles was measured by the following method.

That is, 3 g of the non-magnetic acicular black iron-based composite particles and 40 ml of ethanol were placed in a 50-ml precipitation pipe and then was subjected to ultrasonic dispersion for 20 minutes. Thereafter, the obtained dispersion was allowed to stand for 120 minutes, and separated the carbon black fine particles desorbed from the non-magnetic acicular black iron-based composite particles on the basis of the difference in specific gravity therebetween. Next, the thus separated non-magnetic acicular black iron-based composite particles were mixed again with 40 ml of ethanol, and the obtained mixture was further subjected to ultrasonic dispersion for 20 minutes. Thereafter, the obtained dispersion was allowed to stand for 120 minutes, thereby separating the non-magnetic acicular black iron-based composite particles and carbon black desorbed, from each other. The thus separated non-magnetic acicular black iron-based composite particles were dried at 100°C for one hour, and then the carbon content thereof was measured by the "Horiba Metal, Carbon and Sulfur Analyzer EMIA-2200 Model" (manufactured by HORIBA SEISAKUSHO CO., LTD.). The desorption percentage (T %) was calculated according to the following formula:

$$T (\%) = \{(W_a - W_e)/W_a\} \times 100$$

wherein  $W_a$  represents an amount of carbon black fine particles initially adhered on the non-magnetic acicular black iron-based composite particles; and  $W_e$  represents an amount of carbon black fine particles which still remains adhered on the non-magnetic acicular black iron-based composite particles after the above desorption test.

The closer to zero the desorption percentage (T %), the smaller the amount of carbon black fine particles desorbed from the non-magnetic acicular black iron-based composite particles.

- (10) The <u>viscosity of the coating composition</u> was obtained by measuring the viscosity of the coating composition at 25°C at a shear rate D of 1.92 sec<sup>-1</sup> by using "E type viscometer EMD-R" (manufactured by TOKYO KEIKI, CO., LTD.).
- (11) The gloss of the surface of the coating film of each of the non-magnetic undercoat layer and the magnetic recording layer was measured at an angle of incidence of 45° by "glossmeter UGV-5D" (manufactured by SUGA SHIKENKI, CO., LTD.).
- (12) The <u>surface roughness Ra</u> is expressed by the center-line average roughness of the surface of the coating film by using "Surfcom-575A" (manufactured by TOKYO SEIMITSU CO., LTD.).
  - (13) The <u>strength</u> of the coating film was expressed the Young's modulus obtained by "Autograph" (produced by Shimazu Seisakusho Co., Ltd.). The Young's modulus was expressed by the ratio of the Young's modulus of the coating film to that of a commercially available video tape "AV T-120" (produce by VICTOR COMPANY OF JAPAN, LIMITED). The higher the relative value, the more the strength of the coating film is favorable.
  - (14) The <u>magnetic properties</u> of the magnetic particles and magnetic recording medium were measured under an external magnetic field of 10 kOe by "Vibration Sample Magnetometer VSM-3S-15 (manufactured by TOEI KOGYO, CO., LTD.)".
  - (15) The <u>light transmittance</u> is expressed by the linear adsorption coefficient measured by using "UV-Vis Recording Spectrophotometer UV-2100" (manufactured by SHIMAZU SEISAKUSHO, LTD.). The linear adsorption coefficient is defined by the following formula, and the larger the value, the more the transmittance of light become difficult:

Linear adsorption coefficient 
$$(\mu m^{-1}) = \{I \ n \ (I/t)\}/FT$$

wherein t represents a light transmittance (-) at  $\lambda$  = 900 nm, and FT represents thickness ( $\mu$ m) of the coating composition of the film used for the measurement.

(16) The <u>surface resistivity value of a coating film</u> was measured as follows. The coating film to be measured was exposed to an atmosphere kept at a temperature of 25°C and a relative humidity of 60%, for not less than 12 hours. Thereafter, the coating film was slit into 6 mm width, and then placed on metal electrodes each having a width of 6.5 mm such that a coating surface thereof was contacted with the metal electrodes. Two 170-gram weights were

fitted to opposite ends of the coating film so as to bring the coating film into close contact with the metal electrodes. Then, a D.C. voltage of 500V was applied between the metal electrodes, thereby measuring a surface resistivity value of the coating film.

(17) The <u>thickness</u> of each of the base film, the non-magnetic undercoat layer and the magnetic recording layer constituting the magnetic recording medium was measured in the following manner by using "Digital Electronic Micrometer R351C" (manufactured by ANRITSU CORP.)

The thickness (A) of a base film was first measured. Similarly, the thickness (B) (B = the sum of the thicknesses of the base film and the non-magnetic undercoat layer) of a non-magnetic substrate obtained by forming a non-magnetic undercoat layer on the base film was measured. Furthermore, the thickness (C) (C = the sum of the thicknesses of the base film, the non-magnetic undercoat layer and the magnetic recording layer) of a magnetic recording medium obtained by forming a magnetic recording layer on the non-magnetic substrata was measured. The thickness of the non-magnetic undercoat layer is expressed by (B) - (A), and the thickness of the magnetic recording layer is expressed by (C) - (B).

### Example 1:

5

10

15

(Production of non-magnetic acicular black iron-based composite particles)

[0155] 20 kg of acicular manganese-containing hematite particles obtained according to the method described in Japanese Patent Application Laid-open (KOKAI) No. 7-66020 (1995) and shown in the electron photograph (× 60,000) of Fig. 1 (average major axis diameter: 0.151 μm; average minor axis diameter: 0.0220 μm; aspect ratio: 6.9:1; geometrical standard deviation of major axis diameter: 1.35; BET specific surface area value: 50.9 m²/g; Mn content: 13.3 wt. %; and blackness (L\* value): 29.7; volume resistivity: 7.5 × 10<sup>8</sup> Ω • cm), were diaggregated in 150 liters of pure water using a stirrer, and further passed through a TK pipeline homomixer (manufactured by TOKUSHU KIKA KOGYO CO., LTD.) three times, to obtain a slurry containing the acicular manganese-containing hematite particles.

**[0156]** Successively, the obtained slurry was passed through a transverse-type sand grinder (tradename "MIGHTY MILL MHG-1.5L", manufactured by INOUE SEISAKUSHO CO., LTD.) five times at an axis-rotating speed of 2,000 rpm, thereby obtaining a slurry in which the acicular manganese-containing hematite particles were dispersed.

[0157] The acicular manganese-containing hematite particles in the obtained slurry, which remain on a sieve of 325 meshes (mesh size: 44 μm) was 0 %. The slurry was filtered and washed with water, thereby obtaining a wet cake composed of the acicular manganese-containing hematite particles. The obtained wet cake composed of the acicular manganese-containing hematite particles was dried at 120°C. 11.0 kg of the dried particles were then charged into an edge runner "MPUV-2 Model" (tradename, manufactured by MATSUMOTO CHUZO TEKKOSHO CO., LTD.), and mixed and stirred at 60 Kg/cm for 30 minutes, thereby lightly diaggregating the particles.

[0158] 220 g of methyl triethoxysilane was mixed and diluted with 200 ml of ethanol to obtain a solution of methyl triethoxysilane. The methyl triethoxysilane solution was added to the thus diaggregated acicular manganese-containing hematite particles while operating the edge runner. The acicular manganese-containing hematite particles were continuously mixed and stirred at a linear load of 60 Kg/cm for 60 minutes.

[0159] Next, 550 g of carbon black fine particles as shown in the electron photograph (x 60,000) of Fig. 2 (particle shape: granular shape; average particle size: 0.022 μm; geometrical standard deviation of particle sizes: 1.68; BET specific surface area value: 134 m²/g; and blackness (L\* value): 16.6), were added to the acicular manganese-containing hematite particles coated with methyl triethoxysilane for 10 minutes while operating the edge runner. Further, the particles were continuously mixed and stirred at a linear load of 60 Kg/cm for 60 minutes to adhere the carbon black fine particles on the coating layer composed of methyl triethoxysilane, thereby obtaining non-magnetic acicular black iron-based composite particles.

[0160] The obtained non-magnetic acicular black iron-based composite particles were dried or heat-treated at 105°C for 60 minutes by using a drier. The resultant non-magnetic acicular black iron-based composite particles had an average major axis diameter of 0.151  $\mu$ m, an average minor axis diameter of 0.0222  $\mu$ m and an aspect ratio of 6.8:1 as shown in the electron photograph (x 60,000) of Fig. 3. In addition, the non-magnetic acicular black iron-based composite particles showed a geometrical standard deviation of major axis diameter of 1.35, a BET specific surface area value of 52.5 m<sup>2</sup>/g, a blackness (L\* value) of 19.8, a volume resistivity: 3.3 × 10<sup>4</sup>  $\Omega$  • cm and a desorption percentage of carbon black fine particles: 6.5 %. The amount of a coating organosilane compound produced from methyl triethoxysilane was 0.31 % by weight (calculated as Si). Since no carbon black fine particles were recognized on the electron photograph of Fig. 3, it was confirmed that a whole amount of the carbon black fine particles were adhered on the coating layer composed of the organosilane compound produced from methyl triethoxysilane.

**[0161]** For a comparative purpose, the acicular manganese-containing hematite particles not coated with methyl triethoxysilane and the carbon black fine particles were mixed and stirred together by an edge runner in the same manner

as described above, thereby obtaining treated particles as shown in the electron photograph (× 60,000) of Fig. 4. As shown in Fig. 4, it was recognized that the carbon black fine particles were not adhered on the acicular manganese-containing hematite particles, and the individual particles were present separately.

5 (Production of non-magnetic substrate: Formation of non-magnetic undercoat layer on base film)

[0162] 12 g of the non-magnetic acicular black iron-based composite particles obtained in the above were mixed with a binder resin solution (30 % by weight of vinyl chloride-vinyl acetate copolymer resin having a sodium sulfonate group and 70 % by weight of cyclohexanone) and cyclohexanone, and each of the obtained mixtures (solid content: 72 % by weight) was kneaded by a plast-mill for 30 minutes.

[0163] Each of the thus-obtained kneaded material was charged into a 140 ml-glass bottle together with 95 g of 1.5 mmØ glass beads, a binder resin solution (30 % by weight of polyurethane resin having a sodium sulfonate group and 70 % by weight of a solvent (methyl ethyl ketone: toluene = 1:1)), cyclohexanone, methyl ethyl ketone and toluene, and the obtained mixture was mixed and dispersed by a paint shaker for 6 hours to obtain a non-magnetic coating composition.

**[0164]** The thus-obtained non-magnetic coating composition containing the non-magnetic acicular black iron-based composite particles was as follows:

20		
	Non-magnetic acicular black iron-based composite particles	100 parts by weight
	Vinyl chloride-vinyl acetate copolymer resin having a sodium sulfonate group	10 parts by weight
	Polyurethane resin having a sodium sulfonate group	10 parts by weight
25	Cyclohexanone	44.6 parts by weight
	Methylethyl ketone	111.4 parts by weight
	Toluene	66.9 parts by weight

[0165] The non-magnetic coating composition obtained was applied to a polyethylene terephthalate film of 12  $\mu$ m thick to a thickness of 55  $\mu$ m by an applicator, and the coating film was then dried, thereby forming a non-magnetic undercoat layer. The thickness of the non-magnetic undercoat layer was 3.3  $\mu$ m.

**[0166]** The thus obtained non-magnetic undercoat layer had a gloss of 198 %, and a surface roughness Ra of 6.4 nm. The Young's modulus (relative value) thereof was 121. The linear adsorption coefficient (of the coating film) thereof was  $1.83 \, \mu m^{-1}$ ; and the surface resistivity thereof was  $1.7 \times 10^9 \, \Omega/\text{sg}$ .

## Example 2

30

<u>(Production of magnetic recording medium: Formation of magnetic recording layer)</u>

[0167] 12 g of magnetic acicular metal particles containing iron as a main component (average major axis diameter: 0.115 μm, average minor axis diameter: 0.0182 μm, aspect ratio: 6.3:1, coercive force: 1910 Oe, saturation magnetization: 131 emu/g), 1.2 g of a polishing agent (AKP-30: trade name, produced by SUMITOMO CHEMICAL CO., LTD.), 0.12 g of carbon black (#3250B, trade name, produced by MITSUBISHI CHEMICAL CORP.), a binder resin solution (30% by weight of vinyl chloride-vinyl acetate copolymer resin having a sodium sulfonate group and 70% by weight of cyclohexanone) and cyclohexanone were mixed to obtain a mixture (solid content: 78% by weight). The mixture was further kneaded by a plast-mill for 30 minutes to obtain a kneaded material.

[0168] The thus-obtained kneaded material was charged into a 140 ml-glass bottle together with 95 g of 1.5 mm $\varnothing$  glass beads, a binder resin solution (30 % by weight of polyurethane resin having a sodium sulfonate group and 70 % by weight of a solvent (methyl ethyl ketone : toluene = 1 : 1)), cyclohexanone, methyl ethyl ketone and toluene, and the mixture was mixed and dispersed by a paint shaker for 6 hours. Then, the lubricant and hardening agent were added to the mixture, and the resultant mixture was mixed and dispersed by a paint shaker for 15 minutes.

[0169] The thus-obtained magnetic coating composition was as follows:

5	Magnetic acicular metal particles containing iron as a main component	100 parts by weight
	Vinyl chloride-vinyl acetate copolymer resin having a sodium sulfonate group	10 parts by weight
	Polyurethane resin having a sodium sulfonate group	10 parts by weight
	Polishing agent (AKP-30)	10 parts by weight
10	Carbon black (#3250B)	1.0 parts by weight
	Lubricant (myristic acid: butyl stearate = 1 : 2)	3.0 parts by weight
	Hardening agent (polyisocyanate)	5.0 parts by weight
45	Cyclohexanone	65.8 parts by weight
15	Methyl ethyl ketone	164.5 parts by weight
	Toluene	98.7 parts by weight

- 20 [0170] The magnetic coating composition obtained was applied to the non-magnetic undercoat layer to a thickness of 15 μm by an applicator, and the magnetic recording medium obtained was oriented and dried in a magnetic field, and then calendered. The magnetic recording medium was then subjected to a curing reaction at 60°C for 24 hours, and thereafter slit into a width of 0.5 inch, thereby obtaining a magnetic tape. The thickness of the respective magnetic recording layer was 1.2 μm.
- 25 **[0171]** The coercive force Hc of the magnetic tape produced by forming a magnetic recording layer on the non-magnetic undercoat layer was 2037 Oe, the squareness (Br/Bm) thereof was 0.87, the gloss thereof was 223 %, the surface roughness Ra thereof was 6.3 nm, the Young's modulus (relative value) thereof was 133, the linear absorption coefficient thereof was 2.08 μm<sup>-1</sup>. and the surface resistivity of 1.3 × 10<sup>8</sup> Ω/sq.

### 30 Core particles 1 to 5:

- **[0172]** Various acicular hematite particles and acicular iron oxide hydroxide particle, (goethite) particles produced by known methods, were prepared. The same procedure as defined in Example 1 was conducted by using these particles, thereby obtaining diaggregated acicular hematite particles and acicular iron oxide hydroxide particles.
- [0173] Various properties of the acicular hematite particles and acicular iron oxide hydroxide particles as core particles are shown in Table 1.
  - **[0174]** Meanwhile, the core particles 5 were acicular goethite particles produced by the method described in Japanese Patent Application Laid-open (KOKAI) No. 4-144924(1992). The core particles 3 were acicular hematite particles produced by heat-dehydrating the acicular goethite particles obtained by the method described in Japanese Patent Application Laid-open (KOKAI) No. 4-144924(1992).

## Core particles 6:

- [0175] The same procedure as defined in Example 1 was conducted by using 20 kg of the diaggregated acicular hematite particles (core particles 1) and 150 liters of water, thereby obtaining a slurry containing the acicular hematite particles. The pH value of the obtained redispersed slurry containing the acicular hematite particles was adjusted to 10.5, and then the concentration of the solid content in the slurry was adjusted to 98 g/liter by adding water thereto. After 150 liters of the slurry was heated to 60°C, 5444 ml of a 1.0 mol/liter NaAlO<sub>2</sub> solution (corresponding to 1.0 % by weight (calculated as Al) based on the weight of the acicular hematite particles) was added to the slurry. After allowing the obtained slurry to stand for 30 minutes, the pH value of the slurry was adjusted to 7.5 by using acetic acid. After further allowing the resultant slurry to stand for 30 minutes, the slurry was subjected to filtration, washing with water, drying and pulverization, thereby obtaining the acicular manganese-containing hematite particles whose surface was coated with hydroxides of aluminum.
- [0176] The thus obtained acicular hematite particles whose surface was coated with hydroxides of aluminum had average major axis diameter: 0.143 μm; average minor axis diameter: 0.0210 μm; and aspect ratio: 6.8:1. The geometrical standard deviation of major axis diameter was 1.37, the BET specific surface area value was 54.9 m²/g, the blackness (L\* value) was 28.4, and the volume resistivity was 4.6 × 10<sup>8</sup> Ω cm. As a result of fluorescent X-ray analysis, the obtained particles had an Al content of 0.98 % by weight.

## Core particles 7 to 10:

10

20

25

35

[0177] The same procedure as defined above for the production of the core particles 6, was conducted except that kinds of core particles and kinds and amounts of additives used in the above surface treatment were changed variously, thereby obtaining surface-treated acicular hematite particles and surface-treated acicular iron oxide hydroxide particles.

[0178] The essential treating conditions are shown in Table 2, and various properties of the obtained surface-treated core particles are shown in Table 3.

### Production Examples 1 to 10 and Production Comparative Examples 1 to 5:

[0179] The same procedure as defined in Example 1 was conducted except that kinds of acicular hematite particles and acicular iron oxide hydroxide particles, addition or non-addition of alkoxysilane, kinds and amounts of alkoxysilane added, treating conditions of an edge runner used in the alkoxysilane-coating process, kinds and amounts of carbon black fine particles adhered, and treating conditions of an edge runner used in the process for adhering the carbon black fine particles, were changed variously, thereby obtaining non-magnetic acicular black iron-based composite particles. As a result of the observation by an electron microscope, carbon black fine particles were not recognized in the non-magnetic acicular black iron-based composite particles obtained in Production Examples 1 to 10. Therefore, it was confirmed that a substantially whole amount of the carbon black fine particles used in Production Examples 1 to 10 were adhered on the coating layer composed of an organosilane compound produced from the alkoxysilane.

**[0180]** Various properties of the carbon black fine particles A to C used, are shown in Table 4. The essential treating conditions are shown in Table 5, and various properties of the obtained non-magnetic acicular black iron-based composite particles are shown in Table 6.

## Examples 3 to 12 and Comparative Examples 1 to 13:

(Production of non-magnetic substrate: Formation of non-magnetic undercoat layer on non-magnetic base film)

**[0181]** By using the non-magnetic acicular black iron-based composite particles obtained in Production Example 1 to 10 and Production Comparative Examples 1 to 5, core particles 1 to 5 and carbon black fine particles A to C, non-magnetic undercoat layers were formed in the same way as in Example 1.

[0182] The main producing conditions and various properties are shown in Table 7.

## Examples 13 to 22 and Comparative Examples 14 to 26:

(Production of magnetic recording medium: Formation of magnetic coating film)

**[0183]** Magnetic recording media were produced in the same way as in Example 2 except for varying the kind of non-magnetic undercoat layer and the kind of magnetic particles.

[0184] Various properties of the magnetic particles (1) to (4) used, are shown in Table 8.

© [0185] The main producing conditions shown in Table 9 and various properties are shown in Tables 9 and 10.

## **Production Example 11:**

[0186] 220 g of methyl hydrogen polysiloxane (tradename: "TSF484", produced by TOSHIBA SILICONE CO., LTD.) was added to the diaggregated acicular manganese-containing hematite particles obtained in Example 1, while operating an edge runner "MPUV-2 Model" (manufactured by MATSUMOTO CHUZO TEKKOSHO CO., LTD.), and then continuously mixed and stirred at a linear load of 40 kg/cm for 30 minutes.

[0187] Next, 550 g of carbon black fine particles (particle shape: granular shape; particle size: 0.022 µm; geometrical standard deviation of particle sizes: 1.68; BET specific surface area value: 134 m²/g; and blackness (L\* value): 16.6) were added to the acicular manganese-containing hematite particles coated with methyl hydrogen polysiloxane, for 10 minutes while operating the edge runner. Further, the obtained particles were continuously mixed and stirred at a linear load of 40 kg/cm for 20 minutes to adhere the carbon black fine particles on the coating layer composed of methyl hydrogen polysiloxane, thereby obtaining non-magnetic acicular black iron-based composite particles.

[0188] The obtained non-magnetic acicular black iron-based composite particles were dried at 105°C for 60 minutes by using a drier. The obtained non-magnetic acicular black iron-based composite particles were granular particles having an average major axis diameter of 0.151 μm, an average minor axis diameter of 0.0221 μm and an aspect ratio of 6.8:1. In addition, the geometrical standard deviation of major axis diameter of the non-magnetic acicular black iron-based composite particles was 1.35; the BET specific surface area value was 51.8 m<sup>2</sup>/g; the Mn content was 12.4 %

by weight; the blackness (L\* value) was 18.6; the volume resistivity was  $2.6 \times 10^4 \,\Omega$  • cm; and the carbon black desorption percentage was 6.3 %. The coating amount of the methyl hydrogen polysiloxane was 0.82 % by weight (calculated as Si). As a result of the observation by an electron microscope, since almost carbon black fine particles were not recognized in the obtained composite particles, it was confirmed that substantially a whole amount of the carbon black fine particles were adhered on the coating layer composed of the methyl hydrogen polysiloxane.

Production Examples 12 to 21 Production Comparative Examples 6 to 8:

[0189] The same procedure as defined in Production Example 11 was conducted except that kinds of acicular hematite particles and acicular iron oxide hydroxide particles as the core particles, kinds and amounts of polysiloxane added, treating conditions of an edge runner used in the polysiloxane-coating process, kinds and amounts of carbon black fine particles adhered, and treating conditions of an edge runner used in the process for adhering the carbon black fine particles, were changed variously, thereby obtaining non-magnetic acicular black iron-based composite particles. As a result of the observation by an electron microscope, almost no carbon black fine particles were recognized in the non-magnetic acicular black iron-based composite particles obtained in Production Examples 12 to 21. Therefore, it was confirmed that substantially a whole amount of the carbon black fine particles used were adhered on the coating layer composed of polysiloxane.

**[0190]** The essential treating conditions are shown in Table 11, and various properties of the obtained non-magnetic acicular black iron-based composite particles are shown in Table 12.

Examples 23 to 33 and Comparative Examples 27 to 29:

(Production of non-magnetic substrate: Formation of non-magnetic undercoat layer on non-magnetic base film)

[0191] By using the non-magnetic acicular black iron-based composite particles obtained in Production Example 11 to 21 and Production Comparative Examples 6 to 8, non-magnetic undercoat layers were formed in the same way as in Example 1.

[0192] The main producing conditions and various properties are shown in Table 13.

Examples 34 to 44 and Comparative Examples 30 to 32:

(Production of magnetic recording medium: Formation of magnetic coating film)

**[0193]** Magnetic recording media were produced in the same way as in Example 2 except for varying the kind of non-magnetic undercoat layer and the kind of magnetic particles.

[0194] The main producing conditions and various properties are shown in Table 14.

## **Production Example 22:**

- [0195] 220 g of polyether-modified polysiloxane (tradename "BYK-080", produced by BYK-Chemie CO., LTD.) were added to the diaggregated acicular manganese-containing hematite particles obtained in Example 1 particles 1, while operating an edge runner "MPUV-2 Model" (manufactured by MATSUMOTO CHUZO TEKKOSHO CO., LTD.), and then continuously mixed and stirred at a linear load of 40 kg/cm for 30 minutes.
- [0196] Next, 550 g of carbon black fine particles (particle shape: granular shape; particle size: 0.022 µm; geometrical standard deviation of particle sizes: 1.68; BET specific surface area: 134 m²/g; and blackness (L\* value): 16.6) were added to the acicular manganese-containing hematite particles coated with polyether-modified polysiloxane, for 10 minutes while operating the edge runner. Further, the obtained particles were continuously mixed and stirred at a linear load of 40 kg/cm for 20 minutes to adhere the carbon black fine particles on the coating layer composed of polyether-modified polysiloxane, thereby obtaining non-magnetic acicular black iron-based composite particles.
- [0197] The obtained non-magnetic acicular black iron-based composite particles were dried at 105°C for 60 minutes by using a drier. The obtained non-magnetic acicular black iron-based composite particles had an average major axis diameter of 0.151 μm, an average major axis diameter of 0.0221 μm and an aspect ratio of 6.8:1. In addition, the geometrical standard deviation of major axis diameters of the non-magnetic acicular black iron-based composite particles was 1.35; the BET specific surface area value was 53.1 m²/g; the Mn content was 12.3 % by weight; and the volume resistivity was 3.1 × 10<sup>4</sup> Ω cm; the blackness (L\* value) was 18.5; the carbon black desorption percentage was 5.8 %. The coating amount of the polyether-modified polysiloxane was 0.34 % by weight (calculated as Si). As a result of the observation by an electron microscope, since almost no carbon black fine particles were recognized in the obtained composite particles, it was confirmed that substantially a whole amount of the carbon black fine particles were adhered

on the coating layer composed of the polyether-modified polysiloxane.

## Production Examples 23 to 32 and Production Comparative Examples 9 to 11:

[0198] The same procedure as defined in Production Example 22 was conducted except that kinds of acicular hematite particles and acicular iron oxide hydroxide particles as the core particles, kinds and amounts of modified polysiloxane added, treating conditions of an edge runner used in the modified polysiloxane-coating process, kinds and amounts of carbon black fine particles adhered, and treating conditions of an edge runner used in the process for adhering the carbon black fine particles, were changed variously, thereby obtaining non-magnetic acicular black iron-based composite particles. As a result of the observation by an electron microscope, almost no carbon black fine particles were recognized in the non-magnetic acicular black iron-based composite particles obtained in Production Examples 23 to 32. Therefore, it was confirmed that substantially a whole amount of the carbon black fine particles used were adhered on the coating layer composed of modified polysiloxane.

**[0199]** The essential treating conditions are shown in Table 15, and various properties of the obtained non-magnetic acicular black iron-based composite particles are shown in Table 16.

Examples 45 to 55 and Comparative Examples 33 to 35:

(Production of non-magnetic substrate: Formation of non-magnetic undercoat layer on non-magnetic base film)

**[0200]** By using the non-magnetic acicular black iron-based composite particles obtained in Production Example 22 to 32 and Production Comparative Examples 9 to 11, non-magnetic undercoat layers were formed in the same way as in Example 1

**[0201]** The main producing conditions and various properties are shown in Table 17.

Examples 56 to 66 and Comparative Examples 36 to 38:

(Production of magnetic recording medium: Formation of magnetic coating film)

[0202] Magnetic recording media were produced in the same way as in Example 2 except for varying the kind of non-magnetic undercoat layer and the kind of magnetic particles.

[0203] The main producing conditions and various properties are shown in Table 18.

## Production Example 33:

[0204] 220 g of carboxylic acid-terminal-modified polysiloxane (tradename: "TSF4770", produced by TOSHIBA SILI-CONE CO., LTD.) was added to the diaggregated acicular manganese-containing hematite particles obtained in Example 1, while operating an edge runner "MPUV-2 Model" (manufactured by MATSUMOTO CHUZO TEKKOSHO CO., LTD.), and then continuously mixed and stirred at a linear load of 40 kg/cm for 30 minutes.

[0205] Next, 550 g of carbon black fine particles (particle shape: granular shape; particle size: 0.022 µm; geometrical standard deviation of particle sizes: 1.68; BET specific surface area value: 134 m²/g; and blackness (L\* value): 16.6) were added to the acicular manganese-containing hematite particles coated with carboxylic acid-terminal-modified polysiloxane, for 10 minutes while operating the edge runner. Further, the obtained particles were continuously mixed and stirred at a linear load of 40 kg/cm for 20 minutes to adhere the carbon black fine particles on the coating layer composed of carboxylic acid-terminal-modified polysiloxane, thereby obtaining non-magnetic acicular black iron-based composite particles.

[0206] The obtained non-magnetic acicular black iron-based composite particles were dried at 105°C for 60 minutes by using a drier. The obtained non-magnetic acicular black iron-based composite particles were granular particles having an average major axis diameter of 0.0221  $\mu$ m and an aspect ratio of 6.8:1. In addition, the geometrical standard deviation of major axis diameters of the non-magnetic acicular black iron-based composite particles was 1.35; the BET specific surface area value was 53.4 m²/g; the Mn content was 12.3 % by weight; and the volume resistivity was 2.3 × 10<sup>4</sup>  $\Omega$  • cm; the blackness (L\* value) was 18.7; the carbon black desorption percentage was 7.1 %. The coating amount of the carboxylic acid-terminal-modified polysiloxane was 0.44 % by weight (calculated as Si). As a result of the observation by an electron microscope, since almost no carbon black fine particles were recognized in the obtained composite particles, it was confirmed that substantially a whole amount of the carbon black fine particles were adhered on the coating layer composed of carboxylic acid-terminal-modified polysiloxane.

27

20

25

15

Production Examples 34 to 43 and Production Comparative Examples 12 to 14:

[0207] The same procedure as defined in Production Example 33 was conducted except that kinds of acicular hematite particles and acicular iron oxide hydroxide particles as the core particles, kinds and amounts of terminal-modified polysiloxane added, treating conditions of an edge runner used in the terminal-modified polysiloxane -coating process, kinds and amounts of carbon black fine particles adhered, and treating conditions of an edge runner used in the process for adhering the carbon black fine particles, were changed variously, thereby obtaining non-magnetic acicular black iron-based composite particles. As a result of the observation by an electron microscope, almost no carbon black fine particles were recognized in the non-magnetic acicular black iron-based composite particles obtained in Production Examples 34 to 43. Therefore, it was confirmed that substantially a whole amount of the carbon black fine particles used were adhered on the coating layer composed of terminal-modified polysiloxane.

**[0208]** The essential treating conditions are shown in Table 19, and various properties of the obtained non-magnetic acicular black iron-based composite particles are shown in Table 20.

15 Examples 67 to 77 and Comparative Examples 39 to 41:

(Production of non-magnetic substrate: Formation of non-magnetic undercoat layer on non-magnetic base film)

[0209] By using the non-magnetic acicular black iron-based composite particles obtained in Production Example 33 to 43 and Production Comparative Examples 12 to 14, non-magnetic undercoat layers were formed in the same way as in Example 1. The main producing conditions and various properties are shown in Table 21.

Examples 78 to 88 Comparative Examples 42 to 44:

5 <u>\(\sumsymbol\) Production of magnetic recording medium: Formation of magnetic coating film\)</u>

**[0210]** Magnetic recording media were produced in the same way as in Example 2 except for varying the kind of non-magnetic undercoat layer and the kind of magnetic particles.

[0211] The main producing conditions and various properties are shown in Table 22.

Production Example 44:

30

**[0212]** 220 g of tridecafluorooctyl trimethoxysilane (tradename "TSL8257", produced by TOSHIBA SILICONE CO., LTD.) were added to the diaggregated acicular manganese-containing hematite particles obtained in Example 1, while operating an edge runner "MPUV-2 Model" (manufactured by MATSUMOTO CHUZO TEKKOSHO CO., LTD.), and then continuously mixed and stirred at a linear load of 40 kg/cm for 30 minutes.

**[0213]** Next, 550 g of carbon black fine particles (particle shape: granular shape; particle size: 0.022 µm; geometrical standard deviation of particle sizes: 1.68; BET specific surface area value: 134 m²/g; and blackness (L\* value): 16.6) were added to the acicular manganese-containing hematite particles coated with tridecafluorooctyl trimethoxysilane, for 10 minutes while operating the edge runner. Further, the obtained particles were continuously mixed and stirred at a linear load of 40 kg/cm for 20 minutes to adhere the carbon black fine particles on the coating layer composed of tridecafluorooctyl trimethoxysilane, thereby obtaining non-magnetic acicular black iron-based composite particles.

[0214] The obtained non-magnetic acicular black iron-based composite particles was dried or heat-treated at 105°C for 60 minutes by using a drier. The obtained non-magnetic acicular black iron-based composite particles had an average major axis diameter of 0.151  $\mu$ m, an average major axis diameter of 0.0222  $\mu$ m and an aspect ratio of 6.9:1. In addition, the geometrical standard deviation of major axis diameters of the non-magnetic acicular black iron-based composite particles was 1.35; the BET specific surface area value was 53.2 m²/g; the Mn content was 12.4 % by weight; and the volume resistivity was  $2.7 \times 10^4 \,\Omega$  • cm; the blackness (L\* value) was 18.6; the carbon black desorption percentage was 7.0 %. The coating amount of the fluoroalkyl organosilane compound produced from tridecafluorooctyl trimethoxysilane was 0.13 % by weight (calculated as Si). As a result of the observation by an electron microscope, since almost no carbon black fine particles were recognized in the obtained composite particles, it was confirmed that substantially a whole amount of the carbon black fine particles were adhered on the coating layer composed of the fluoroalkyl organosilane compound produced from tridecafluoroalkyl trimethoxysilane.

55 Production Examples 45 to 54 and Production Comparative Examples 15 to 17:

[0215] The same procedure as defined in Production Example 44 was conducted except that kinds of acicular hematite particles and acicular iron oxide hydroxide particles as the core particles, kinds and amounts of fluoroalkyl silane

compounds added, treating conditions of an edge runner used in the fluoroalkyl silane compound-coating process, kinds and amounts of carbon black fine particles adhered, and treating conditions of an edge runner used in the process for adhering the carbon black fine particles, were changed variously, thereby obtaining non-magnetic acicular black iron-based composite particles. As a result of the observation by an electron microscope, almost no carbon black fine particles were recognized in the non-magnetic acicular black iron-based composite particles obtained in Production Examples 45 to 54. Therefore, it was confirmed that substantially a whole amount of the carbon black fine particles used were adhered on the coating layer composed of fluoroalkyl organosilane compounds.

**[0216]** The essential treating conditions are shown in Table 23, and various properties of the obtained non-magnetic acicular black iron-based composite particles are shown in Table 24.

Examples 89 to 99 and Comparative Examples 45 to 47:

10

30

35

40

45

50

55

(Production of non-magnetic substrate: Formation of non-magnetic undercoat layer on non-magnetic base film)

[0217] By using the non-magnetic acicular black iron-based composite particles obtained in Production Examples 44 to 54 and Production Comparative Examples 15 to 17, non-magnetic undercoat layers were formed in the same way as in Example 1.

[0218] The main producing conditions and various properties are shown in Table 25.

20 Examples 100 to 110 and Comparative Examples 48 to 50:

(Production of magnetic recording medium: Formation of magnetic coating film)

**[0219]** Magnetic recording media were produced in the same way as in Example 2 except for varying the kind of non-magnetic undercoat layer and the kind of magnetic particles.

[0220] The main producing conditions and various properties are shown in Table 26.

Table 1

Core particles	Properties of ac particles or aci hydroxide	cular iron oxide
	Kind	Particle shape
Core particles	Hematite particles	Acicular
Core particles	Hematite particles	Spindle-shaped
Core particles	Mn-containing hematite particles	Acicular
Core particles	Goethite particles	Acicular
Core particles 5	Mn-containing goethite particles	Acicular

Table 1 (continued)

	· · · · · · · · · · · · · · · · · · ·			
	Prope	rties of a	cicular	hematite
	partic	cles or ac	icular in	con oxide
	_	hydroxide		
	Average	Average	Aspect	Geometrical
Core particles	major	minor	ratio	standard
1	1			
	axis	axis	( – )	deviation
	diameter	diameter		(-)
	(mm)	(µm)		
Core particles	0.143	0.0210	6.8:1	1.38
1				
Core particles	0.187	0.0240	7.8:1	1.33
2				
Core particles	0.158	0.0211	7.5:1	1.43
3				
Core particles	0.240	0.0272	8.8:1	1.37
4				
Core particles	0.189	0.0220	8.6:1	1.41
5				

Mn

content

(wt. %)

9.0

8.1

BET

specific

surface

area

 $(m^2/g)$ 

55.3

43.3

53.6

86.3

110.6

Core

particles

Core

particles 1

Core

particles 2

Core

particles 3

Core

particles 4

Core

particles 5

## Table 1 (continued)

Properties of acicular hematite particles or acicular iron oxide hydroxide particles

Volume

resistivity

 $(\Omega \cdot cm)$ 

 $2.3 \times 10^{8}$ 

 $8.6 \times 10^{8}$ 

 $2.6 \times 10^{7}$ 

 $x 10^{7}$ 

 $\times 10^{7}$ 

3.2

9.6

Blackness

(L\*

value)

(-)

28.3

32.6

25.4

34.6

28.3

5

10

15

20

25

Table 2

30

35

40

45

50

Kind of Surface-treating process Additives Core core particles particles Kind Calcu-Amount lated as (wt. %) Core Core Sodium Al 1.0 particles 6 aluminate particles 1 #3 Water 0.5 Core Core  $SiO_2$ particles 7 particles glass 2 Core Core Aluminum Al 1.5 sulfate particles 8 particles 3 #3 Water  $SiO_2$ 0.5 glass Core Sodium Αl 1.0 Core particles 9 particles aluminate Colloidal  $SiO_2$ 3.0 4 silica Core Aluminum Al 5.5 Core particles acetate particles 10

# Table 2 (continued)

Core   Coating material	5		Surfa	ce-treating pr	ocess
Particles   Rind   Calculated   Amount     as		Core	C	oating materia	1
Core particles 6 A Al 0.98  Core particles 7 S SiO2 0.48  Core particles 8 A Al 1.47  SiO2 0.46  Core particles 9 A Al 0.96  SiO2 2.81  Core particles A Al 5.21	10	particles	Kind	Calculated	Amount
Core particles 7 S SiO <sub>2</sub> 0.48  Core particles 8 A Al 1.47  SiO <sub>2</sub> 0.46  Core particles 9 A Al 0.96  SiO <sub>2</sub> 2.81  Core particles A Al 5.21				as	(wt. %)
Core particles 7 S SiO <sub>2</sub> 0.48  Core particles 8 A Al 1.47  SiO <sub>2</sub> 0.46  Core particles 9 A Al 0.96  SiO <sub>2</sub> 2.81  Core particles A Al 5.21	15	Core particles 6	Α	Al	0.98
S SiO <sub>2</sub> 0.46  Core particles 9 A Al 0.96  S SiO <sub>2</sub> 2.81  Core particles A Al 5.21		Core particles 7	S	SiO <sub>2</sub>	0.48
S   SiO <sub>2</sub>   0.46     Core particles 9   A   Al   0.96     S   SiO <sub>2</sub>   2.81     Core particles   A   Al   5.21		Core particles 8	A	Al	1.47
25 SiO <sub>2</sub> 2.81  Core particles A Al 5.21	20		S	SiO <sub>2</sub>	0.46
Core particles A Al 5.21		Core particles 9	A	Al	0.96
	25		S	SiO <sub>2</sub>	2.81
10		Core particles	A	Al	5.21
		10			

Note; A: Hydroxide of aluminum S: Oxide of silicon

Table 3

	Core particles	Properties of surface-treated core particles				
5		Average major axis diameter (µm)	Average minor axis diameter (µm)	Aspect ratio (-)	Geometrical standard deviation (-)	
	Core particles 6	0.143	0.0210	6.8:1	1.37	
10	Core particles 7	0.187	0.0241	7.8:1	1.33	
, 0	Core particles 8	0.158	0.0212	7.5:1	1.43	
	Core particles 9	0.239	0.0271	8.8:1	1.36	
	Core particles 10	0.189	0.0220	8.6:1	1.41	
15		BET specific surface area (m²/g)	Mn content (wt. %)	Volume resistivity (Ω • cm)	Blackness (L* value) (-)	
	Core particles 6	54.9	-	4.6 x 10 <sup>8</sup>	28.4	
	Core particles 7	43.6	-	9.1 x 10 <sup>8</sup>	33.1	
20	Core particles 8	53.5	8.8	4.2 x 10 <sup>7</sup>	25.8	
	Core particles 9	85.8	-	9.3 x 10 <sup>7</sup>	35.0	
	Core particles 10	111.3	7.6	4.8 x 10 <sup>7</sup>	28.5	

	Iable 4				
30	Kind of carbon black fine particles	Properties of carbon black fine particles			
		Particle shape	Average major axis diameter (µm)	Geometrical standard deviation (-)	
35	Carbon black A	Granular	0.022	1.78	
	Carbon black B	Granular	0.015	1.56	
	Carbon black C	Granular	0.030	2.06	
40		BET specific surface area (m2/g)	Oil absorption (ml/100g)	Blackness (L* value) (-)	
	Carbon black A	133.5	84	14.6	
	Carbon black B	265.3	57	15.2	
45	Carbon black C	84.6	95	17.0	

# Table 5

		<u></u>		
5			Production of non	
· ·	Production		acicular black i	ron-based
	Examples	Kind of core	composite part	cicles
	and	particles	Addition of alkoxy	silane or
	Production		silicon comp	
10	Comparative		Additives	5
	Examples		Kind	Amount
				added (part
				by weight)
	Production	Core	Methyl	1.0
15	Example 1	particles 1	triethoxysilane	
	Production	Core	Methyl	1.5
	Example 2	particles 2	trimethoxysilane	
	Production	Core	Dimethyl	3.5
20	Example 3	particles 3	dimethoxysilane	
	Production	Core	Phenyl	1.0
	Example 4	particles 4	triethoxysilane	:
	Production	Core	Isobutyl	5.0
25	Example 5	particles 5	trimethoxysilane	
20	Production	Core	Methyl	2.0
	Example 6	particles 6	triethoxysilane	
	Production	Core	Methyl	1.0
	Example 7	particles 7	trimethoxysilane	
30	Production	Core	Dimethyl	2.0
	Example 8	particles 8	dimethoxysilane	
	Production	Core	Phenyl	4.5
	Example 9	particles 9	triethoxysilane	
35	Production	Core	Isobutyl	3.0
	Example 10	particles 10	trimethoxysilane	
	Production	Core	<del>-</del>	_
	Comparative	particles 1		
	Example 1			
40	Production	Core	Methyl	1.0
	Comparative	particles 1	triethoxysilane	
	Example 2			
	Production	Core	Dimethyl	0.5
45	Comparative	particles 4	dimethoxysilane	
	Example 3			
	Production	Core	Methyl	0.005
	Comparative	particles 4	triethoxysilane	
	Example 4		1	
50	Production	Core	aminopropyl	1.0
l	Comparative	particles 1	triethoxysilane	
l	Example 5			

# Table 5 (continued)

5			n of non-magne	
	Production Examples	black iro	n-based composi	te particles
	and	Addition	of alkoxysilan	e or silicon
	Production		compound	
10	Comparative	Edge runne	er treatment	Amount
	Examples			coated
		Linear	Time	(calculated
		load	(min)	as Si)
15		(Kg/cm)		(wt. %)
	Production	60	30	0.16
	Example 1			
	Production	60	60	0.30
	Example 2			
20	Production	75	45	0.79
	Example 3			
	Production	60	30	0.12
	Example 4			
25	Production	45	60	0.75
20	Example 5			
	Production	60	45	0.31
	Example 6			
	Production	50	30	0.20
30	Example 7			
	Production	60	60	0.46
	Example 8			
	Production	75	30	0.50
<i>35</i>	Example 9			
35	Production	60	30	0.46
	Example 10			
	Production	-	-	-
	Comparative			
40	Example 1			0 150
	Production	60	30	0.156
	Comparative			
	Example 2			0.116
.=	Production	60	60	0.116
45	Comparative			
	Example 3		20	7 0 10 4
	Production	60	30	$7.9 \times 10-4$
	Comparative			
50	Example 4			0.126
	Production	60	<del></del>	0.120
	Comparative			
	Example 5			<u> </u>

Table 5 (continued)

	Description	D 7		
5	Production	Production of non-magnetic acicular black iron-based composite particles		
	Examples			
	and		black fine particles	
	Production		fine particles	
10	Comparative	Kind	Amount added	
	Examples		(part by weight)	
	Production	A	5.0	
	Example 1			
15	Production	A	10.0	
15	Example 2			
	Production	В	1.0	
	Example 3			
	Production	В	5.0	
20	Example 4			
	Production	С	5.0	
	Example 5			
	Production	A	5.0	
<i>25</i>	Example 6			
	Production	A	10.0	
	Example 7			
	Production	В	1.0	
	Example 8			
30	Production	В	5.0	
	Example 9	·		
	Production	С	5.0	
	Example 10			
<i>35</i>	Production	A	5.0	
	Comparative			
	Example 1			
	Production	_	-	
40	Comparative			
40	Example 2			
	Production	A	0.01	
	Comparative			
	Example 3			
<b>4</b> 5	Production	В	2.0	
	Comparative	-	· ·	
	Example 4			
	Production	С	5.0	
50	Comparative			
	Example 5			

#### Table 5 (continued)

Production	Production of non-magnetic acicular black iron-based composite particles				
Examples and		carbon black f			
Production		er treatment	Amount		
Comparative	Euge runne	er creatment	adhered		
Examples	Linear load	Time	(calculated		
DAGMPICS	(Kg/cm)	(min)	as C)		
	(Rg/Cm/	(111211)	(wt. %)		
Production	60	60	4.75		
Example 1	60	60	4.75		
Production	45	30	9.09		
Example 2	40	30	J. 05		
Production	60	60	0.99		
Example 3	00	0	0.55		
Production	75	45	4.76		
Example 4					
Production	60	50	4.72		
Example 5					
Production	45	60	4.75		
Example 6					
Production	60	45	9.01		
Example 7					
Production	75	60	0.99		
Example 8					
Production	60	30	4.81		
Example 9 Production					
Example 10	45	60	4.77		
Production	60	30	4.76		
Comparative		30	4.70		
Example 1					
Production	_		<del>-</del>		
Comparative					
Example 2					
Production	45	60	0.009		
Comparative					
Example 3					
Production	60	45	1.96		
Comparative					
Example 4					
Production	<b>4</b> 5	60	4.78		
Comparative					
Example 5					

### Table 6

				. · · · · · · · · · · · · · · · · · · ·	
5	Production				cular black
	Examples		n-based comp		
	and	Average	Average	Aspect	Geometrical
	Production	major	minor	ratio	standard
10	Comparative	axis	axis	( – )	deviation
	Examples	diameter	diameter		(-)
		(µm)	(µm)		
	Production	0.143	0.0211	6.8:1	1.37
15	Example 1				
,0	Production	0.187	0.0242	7.7:1	1.34
	Example 2				
	Production	0.157	0.0211	7.4:1	1.43
	Example 3 Production				
20	Example 4	0.240	0.0273	8.8:1	1.35
	Production				
	Example 5	0.190	0.0221	8.6:1	1.42
	Production				
25	Example 6	0.143	0.0212	6.7:1	1.37
	Production				4 24
	Example 7	0.186	0.0243	7.7:1	1.34
	Production	0 150	0 0010	1	1.43
30	Example 8	0.158	0.0212	7.5:1	1.43
	Production	0.240	0.0273	8.8:1	1.36
	Example 9	0.240	0.0273	0.0:1	1.30
	Production	0.190	0.0222	8.6:1	1.41
	Example 10	0.190	0.0222	0.0.1	7.47
35	Production	0.143	0.0211	6.8:1	1.38
	Comparative				
	Example 1				
	Production	0.143	0.0210	6.8:1	1.38
40	Comparative				
	Example 2				
	Production	0.240	0.0272	8.8:1	1.37
	Comparative				
45	Example 3				
	Production	0.240	0.0273	8.8:1	1.36
	Comparative				
;	Example 4				
50	Production	0.144	0.0212 ,	6.8:1	1.37
50	Comparative				
	Example 5				

55

# Table 6 (continued)

<b>7</b>	Propertie		n-magnetic a		lack iron
Examples		T	composite p		
and	BET	Mn	Volume	Black-	Carbon
Comparative	specific	content	resisti-	ness	black
Examples	surface	(wt. %)	vity	(上*	desorp-
	area		(Ω•cm)	value)	tion
	$(m^2/g)$			( – )	percen-
	, , ,				tage (%
Production			2 5 101		
Example 1	55.6	-	$3.6 \times 10^4$	19.6	7.1
Production			_		
	46.8	_	$8.6 \times 10^{2}$	18.3	6.6
Example 2					
Production	54.1	8.2	$9.6 \times 10^{5}$	18.1	8.3
Example 3					
Production	88.1		$5.8 \times 10^{3}$	20.3	9.1
Example 4	00.1	_	] 3.0 \ 10-	20.5	] ,,,
Production	115 6	6.0	0.6.103	10.0	<u> </u>
Example 5	115.6	6.9	$8.6 \times 10^{3}$	18.9	6.8
Production		<u> </u>	_		
	55.3	-	$5.8 \times 10^{7}$	19.8	4.8
Example 6					
Production	47.2	_	$1.1 \times 10^{3}$	17.9	4.7
Example 7					
Production	53.9	8.0	$3.6 \times 10^{7}$	18.3	4.5
Example 8	55.5	0.0	. 3.0 x 10	10.5	1 4.5
Production	06.0		1 0 105	10 3	2.0
Example 9	86.9	-	$1.8 \times 10^{5}$	19.3	3.2
Production					
	113.1	7.1	$5.1 \times 10^{3}$	19.1	2.9
Example 10					
Production	59.0	_	$1.6 \times 10^{6}$	23.8	63.2
Comparative					
Example 1					
Production	55.2	_	$5.6 \times 10^{7}$	28.6	
Comparative		_	J. 0 X 10	20.0	_
Example 2					
Production	00 1		0 4 107	20.5	İ
Comparative	88.1	-	$8.4 \times 10^{7}$	33.3	-
Example 3					
Production	89.8	-	$9.6 \times 10^{6}$	29.6	41.6
Comparative					
Example 4					
Production	56.7	_	$4.1 \times 10^{6}$	25.2	37.1
Comparative	/ ۵۰۰		4.1 Y TO.	43.4	] 3/.1
Example 5					
-many it			L		l

### Table 7

	n J	tion of	Described of
Examples		Properties of coating	
and	non-magne	composition	
Comparative		sition	
Examples	Kind of non-	Weight ratio	Viscosity
	magnetic	of particles	(cP)
	particles	to resin	
		( - )	
Example 3	Pro. Ex. 1	5.0	410
Example 4	Pro. Ex. 2	5.0	384
Example 5	Pro. Ex. 3	5.0	461
Example 6	Pro. Ex. 4	5.0	1,305
Example 7	Pro. Ex. 5	5.0	1,856
Example 8	Pro. Ex. 6	5.0	358
Example 9	Pro. Ex. 7	5.0	358
Example 10	Pro. Ex. 8	5.0	435
Example 11	Pro. Ex. 9	5.0	1,024
Example 12	Pro. Ex. 10	5.0	1,536
Comparative	Core	5.0	435
Example 1	particles 1	3.0	133
Comparative	Core	5.0	310
Example 2	particles 2	3.0	310
Comparative	Core	5.0	486
Example 3	particles 3	5.0	400
Comparative	Core	5.0	1,126
Example 4	particles 4	3.0	1,120
Comparative	Core	5.0	2,355
Example 5	particles 5	3.0	2,333
Comparative	Carbon black	5.0	19,456
Example 6	A A	3.0	17,450
Comparative	Carbon black	5.0	25,600
Example 7	B Earboil black	3.0	23,000
Comparative	Carbon black	5.0	11,520
· -		3.0	11,520
Example 8 Comparative	C Com	5.0	1,203
	Pro.Com.	٥.٠	1,203
Example 9	Ex. 1	5.0	384
Comparative	Pro.Com.	3.0	204
Example 10 Comparative	Ex. 2 Pro.Com.	15.0	1,306
Example 11	Ex. 3	٠.٥	1,300
Comparative	····	5.0	4,224
Example 12	Pro.Com. Ex. 4	]	3,223
Comparative	Pro.Com.	5.0	1,638
Example 13	Ex. 5	]	1,030
Evambre 13	EX. 3	<u> </u>	<u> </u>

### Table 7 (continued)

Examples	Propertie	s of non-ma	gnetic under	coat layer
and	Thickness	Gloss	Surface	Young's
Comparative	(µm)	(%)	roughness	modulus
Examples			Ra	(relative
			(nm)	value)
Example 3	3.5	205	6.8	121
Example 4	3.4	198	7.2	125
Example 5	3.5	191	8.4	121
Example 6	3.5	186	9.0	128
Example 7	3.5	181	9.6	121
Example 8	3.4	211	6.2	122
Example 9	3.4	201	6.4	126
Example 10	3.5	195	7.8	123
Example 11	3.5	188	9.0	131
Example 12	3.5	185	9.2	123
Comparative	3.5	198	7.0	121
Example 1				
Comparative	3.4	193	8.2	123
Example 2				
Comparative	3.5	186	9.2	120
Example 3				
Comparative	3.5	180	12.1	126
Example 4				
Comparative	3.6	171	14.2	121
Example 5				
Comparative	4.4	36	386	51
Example 6				
Comparative	5.6	18	582	36
Example 7				
Comparative	3.8	68	168	78
Example 8				
Comparative	3.6	118	63.5	111
Example 9				
Comparative	3.5	186	8.8	121
Example 10				
Comparative	3.5	178	11.2	119
Example 11				
Comparative	3.6	156	17.8	115
Example 12				
Comparative	3.7	136	25.6	116
Example 13				

### Table 7 (continued)

5	Examples and	Properties of non-magnetic undercoat layer		
	Comparative	Linear absorption	Surface resistivity	
	Examples	(µm <sup>-1</sup> )	$(\Omega/\mathrm{sq})$	
	Example 3	1.51	8.6 x 10 <sup>9</sup>	
10	Example 4	1.58	4.6 x 10 <sup>9</sup>	
	Example 5	2.16	$1.8 \times 10^9$	
	Example 6	1.46	$2.1 \times 10^{10}$	
	Example 7	1.68	$2.6 \times 10^9$	
15	Example 8	1.64	$7.6 \times 10^9$	
	Example 9	1.81	9.6 x 10 <sup>9</sup>	
	Example 10	2.36	$6.3 \times 10^9$	
	Example 11	1.51	$1.3 \times 10^{10}$	
20	Example 12	1.46	$9.4 \times 10^9$	
	Comparative	0.98	$5.8 \times 10^{13}$	
	Example 1			
	Comparative	1.01	$1.1 \times 10^{14}$	
25	Example 2			
	Comparative	1.21	$5.6 \times 10^{12}$	
	Example 3			
	Comparative	0.78	$1.6 \times 10^{13}$	
30	Example 4	1 10	0.2.1012	
	Comparative Example 5	1.18	$2.3 \times 10^{12}$	
	Comparative		_	
	Example 6			
35	Comparative	-	_	
	Example 7			
	Comparative	-	_	
	Example 8			
40	Comparative	1.28	$5.8 \times 10^{11}$	
	Example 9			
	Comparative	0.92	$7.8 \times 10^{13}$	
	Example 10			
45	Comparative	0.98	$9.6 \times 10^{12}$	
70	Example 11			
	Comparative	1.18	$2.1 \times 10^{11}$	
	Example 12			
50	Comparative	1.28	$1.8 \times 10^{11}$	
50	Example 13	<u> </u>		

Table 8

ť	5		

### 

		Properties	of magnetic
Magnetic	Kind	particles	
particles			
		Average	Average
		major axis	minor axis
		diameter	diameter
		(mm)	(mm)
Magnetic	Magnetic metal	0.127	0.0177
particles	particles containing		
(1)	iron as a main	,	
	component		
Magnetic	Magnetic metal	0.105	0.0148
particles	particles containing		
(2)	iron as a main		
	component		
Magnetic	Co-coated magnetite	0.151	0.0221
particles	particles		
(3)	(Co content: 4.82		
	wt%)		
Magnetic	Co-coated maghemite	0.211	0.0285
particles	particles		
(4)	(Co content: 4.21		
	wt%)		

### Table 8 (continued)

Magnetic particles	Properties of magnetic particles				
	Aspect ratio (-)	Geometrical standard deviation (-	Coercive force Hc (Oe)		
Magnetic particles (1)	7.2:1	1.39	1,915		
Magnetic particles (2)	7.1:1	1.36	1,680		
Magnetic particles (3)	6.8:1	1.44	913		
Magnetic particles (4)	7.4:1	1.36	845		

Saturation

magnetization
 (emu/g)

135.6

128.3

81.3

78.9

### Table 8 (continued)

Properties of magnetic particles

pH value

( - )

9.5

9.9

8.8

8.3

5

10

Magnetic particles

Magnetic

particles (1)

Magnetic

particles (2)

Magnetic

particles (3)

Magnetic

particles (4)

15

20

25

30

35

40

45

50

#### Table 9

5	
10	
15	
20	
25	
30	

40

35

45

55

50

Production of Examples magnetic recording medium Kind of non-Kind of Weight ratio magnetic magnetic of particles undercoat particles to resin layer (-)Example 13 Particles used Example 3 5.0 in Example 2 Example 14 Example 4 Magnetic 5.0 particles (1) Example 15 Example 5 Magnetic 5.0 particles (1) Example 16 5.0 Example 6 Magnetic particles (2) Magnetic Example 17 Example 7 5.0 particles (2) Example 18 Example 8 Particles used 5.0 in Example 2 Example 19 Magnetic 5.0 Example 9 particles (1) 5.0 Example 20 Example 10 Magnetic particles (2) Example 21 Magnetic 5.0 Example 11 particles (3) Example 22 Example 12 Magnetic 5.0 particles (4)

### Table 9 (continued)

Examples	Properties of magnetic recording medium			
Examples	Thickness	Coercive	Br/Bm	Gloss
	of	force Hc	(-)	(%)
	magnetic	(0e)		
	coating			
	film			
	(mm)			
Example 13	1.1	1,970	0.87	226
Example 14	1.1	1,984	0.87	228
Example 15	1.0	1,978	0.86	216
Example 16	1.0	1,768	0.88	215
Example 17	1.1	1,770	0.87	209
Example 18	1.1	1,981	0.87	231_
Example 19	1.1	1,992	0.87	226_
Example 20	1.1	1,780	0.89	219
Example 21	1.0	978	0.90	192
Example 22	1.1	912	0.91	191

### Table 9 (continued)

Properties of

magnetic recording medium

5	

Examples

	Surface	ace Young's Linear		Surface
	roughness	modulus	absorption	resistivity
	Ra	(relative	$(\mu m^{-1})$	$(\Omega/ exttt{sq})$
	(nm)	value)		
Example 13	6.2	125	1.86	$3.2 \times 10^8$
Example 14	6.4	128	1.92	$4.6 \times 10^{8}$
Example 15	7.2	124	2.46	1.9 x 10 <sup>8</sup>
Example 16	6.8	130	1.68	$2.6 \times 10^9$
Example 17	7.4	126	2.01	$8.9 \times 10^{8}$
Example 18	6.0	126	1.91	6.8 x 10 <sup>8</sup>
Example 19	6.4	131	2.12	$3.6 \times 10^{8}$
Example 20	6.2	126	2.71	$2.1 \times 10^{8}$
Example 21	7.2	136	1.83	4.6 x 10 <sup>9</sup>
Example 22	6.8	127	1.61	1.1 x 10 <sup>9</sup>

### Table 10

5								
5			Production of					
	Comparative	magnetic recording medium						
	Examples		<b>Y</b>					
		Kind of non-	Kind of	Weight ratio				
10		magnetic	magnetic	of particles				
		undercoat	particles	to resin				
		layer		( - )				
	Comparative	Comparative	Particles used	5.0				
15	Example 14	Example 1	in Example 2					
10	Comparative	Comparative	Particles used	5.0				
	Example 15	Example 2	in Example 2					
	Comparative	Comparative	Particles used	5.0				
	Example 16	Example 3	in Example 2					
20	Comparative	Comparative	Particles used	5.0				
	Example 17	Example 4	in Example 2					
	Comparative	Comparative	Particles used	5.0				
	Example 18	Example 5	in Example 2					
25	Comparative	Comparative	Particles used	5.0				
	Example 19	Example 6	in Example 2					
	Comparative	Comparative	Particles used	5.0				
	Example 20	Example 7	in Example 2					
	Comparative	Comparative	Magnetic	5.0				
30	Example 21	Example 8	particles (1)					
	Comparative	Comparative	Magnetic	5.0				
	Example 22	Example 9	particles (1)					
	Comparative	Comparative	Magnetic	5.0				
35	Example 23	Example 10	particles (1)					
	Comparative	Comparative	Magnetic	5.0				
	Example 24	Example 11	particles (1)					
	Comparative	Comparative	Magnetic	5.0				
40	Example 25	Example 12	particles (1)					
10	Comparative	Comparative	Magnetic	5.0				
	Example 26	Example 13	particles (1)					
•			- <del>-</del>					

## Table 10 (continued)

5	
10	
15	
20	
25	
30	
35	
40	

		<del></del>		<del></del>		
Q	Properties of					
Comparative Examples	magnetic recording medium					
TYOMPIES	Thickness	Coercive	Br/Bm	Gloss		
	of	force Hc	(-)	(%)		
	magnetic	(0e)	<b>、</b>	( ) /		
	coating	(/				
	film	]				
	(mm)					
Comparative	1.2	1,973	0.87	223		
Example 14						
Comparative	1.2	1,971	0.87	221		
Example 15			·			
Comparative	1.3	1,980	0.86	216		
Example 16						
Comparative	1.1	1,972	0.84	208		
Example 17				·		
Comparative	1.2	1,968	0.83	198		
Example 18						
Comparative	1.3	1,931	0.76	82		
Example 19						
Comparative	1.1	1,928	0.72	68		
Example 20		4 2 2 2				
Comparative	1.3	1,951	0.79	93		
Example 21		1 050		155		
Comparative	1.1	1,958	0.83	156		
Example 22	1 0	1 076		106		
Comparative	1.0	1,976	0.87	196		
Example 23	1.1	1 072	0.00	102		
Comparative	1.1	1,973	0.86	193		
Example 24	1.1	1 060	0.84	165		
Comparative	1.4	1,968	0.84	165		
Example 25	1.1	1,961	0.82	148		
Comparative	4.1	1,301	0.82	148		
Example 26	<u> </u>	<u> </u>		<u></u>		

## Table 10 (continued)

Comparative Examples	Properties of magnetic recording medium					
	Surface	Young's	Linear	Surface		
	roughness	modulus	absorption	resistivity		
	Ra	(relative	(µm <sup>-1</sup> )	$(\Omega/ ext{sq})$		
	(nm)	value)				
Comparative Example 14	6.4	125	1.21	1.8 x 10 <sup>11</sup>		
Comparative Example 15	6.6	127	1.22	8.6 x 10 <sup>11</sup>		
Comparative Example 16	7.2	123	1.39	6.9 x 10 <sup>10</sup>		
Comparative Example 17	11.6	128	1.08	4.6 x 10 <sup>11</sup>		
Comparative Example 18	12.6	123	1.35	$9.8 \times 10^{10}$		
Comparative Example 19	128.0	61	-	-		
Comparative Example 20	284.0	50	-	-		
Comparative Example 21	84.1	121	-	-		
Comparative Example 22	38.2	113	1.44	6.3 x 10 <sup>10</sup>		
Comparative Example 23	7.6	125	1.20	$7.1 \times 10^{10}$		
Comparative Example 24	9.8	123	1.25	$4.1 \times 10^{11}$		
Comparative Example 25	14.4	119	1.39	$9.6 \times 10^{10}$		
Comparative Example 26	18.6	120	1.43	6.0 x 10 <sup>10</sup>		

### Table 11

	Production	<del></del>	Production o	£ 202
5				
	Examples and	Kind of core	magnetic acicul	
	Production	particles	iron-based con	_
		particles	particle	:5
10	Comparative		Addition of mol	
	Examples		Addition of poly Additive	
			Kind	
			Kind	Amount
15				added
15				(part by
	Production	Como montiples 1	TSF484	weight)
j	Example 12	Core particles 1	151404	1.0
	Production	Core particles 2	TSF484	2.0
20	Example 13	Core particles 2	121404	2.0
	Production	Core particles 3	KF99	3.0
	Example 14	Core particles 3	C 7.7	3.0
	Production	Core particles 4	L-9000	5.0
25	Example 15	core particles 4	סטע ת	3.0
	Production	Core particles 5	TSF484/TSF451	1.0/2.0
	Example 16	core parereres 5	101 101/ 101 101	1.0/2.0
	Production	Core particles 6	TSF484	1.5
30	Example 17	COLO PULLUZGO C		
	Production	Core particles 7	TSF484	2.5
	Example 18	•		
	Production	Core particles 8	KF99	4.5
05	Example 19	_		
35	Production	Core particles 9	L-9000	1.0
	Example 20			
	Production	Core particles 10	TSF484/TSF451	2.0/1.0
	Example 21			
40	Production	Core particles 1	TSF484	1.0
	Comparative			
	Example 6			
	Production	Core particles 4	TSF484	0.5
45	Comparative			
	Example 7			
	Production	Core particles 4	TSF484	0.005
	Comparative		1	
50	Example 8			
50				

## Table 11 (continued)

Production	Production of non-magnetic acicular black					
Examples	iron-based composite particles					
and	Addi	tion of polysi	loxane			
Production		er treatment	Amount coated			
Comparative	Linear load	Time	(calculated			
_						
Examples	(Kg/cm)	(min)	as Si)			
			(wt. %)			
Production	40	30	0.44			
Example 12						
Production	60	20	0.84			
Example 13						
Production	20	30	1.23			
Example 14		7.1.				
Production	60	20	2.01			
Example 15						
Production	40	30	1.23			
Example 16						
Production	35	30	0.63			
Example 17			4 3 3			
Production	75	20	1.00			
Example 18		20	1 04			
Production	60	30	1.84			
Example 19	40	2.0	0.40			
Production Example 20	40	30	0.40			
Production	60	30	1.21			
Example 21	80	30	1.21			
Production	60	30	0.42			
Comparative		30	0.42			
Example 6						
Production	60	30	0.21			
Comparative						
Example 7						
Production	60	30	$2.2 \times 10^{-3}$			
Comparative						
Example 8						

### Table 11 (continued)

5	Production	Production of non-magnetic acicular black				
	Examples	iron-based com	posite particles			
	and	Addition of carbon	black fine particles			
10	Production	Carbon black	fine particles			
	Comparative	Kind	Amount added			
	Examples	:	(part by weight)			
15	Production	A	5.0			
,0	Example 12	Α	3.0			
	Production	A	3.0			
	Example 13					
20	Production	В	10.0			
20	Example 14					
	Production	В	7.5			
25	Example 15					
	Production	С	5.0			
	Example 16 Production					
	Example 17	A	5.0			
	Production					
	Example 18	A	2.0			
30	Production	В	3.5			
	Example 19	В				
	Production	В	9.0			
	Example 20		7.0			
35	Production	С	4.5			
	Example 21					
	Production	_	_			
40	Comparative					
	Example 6					
	Production	A	0.01			
	Comparative					
	Example 7 Production					
45	Comparative	В	3.0			
	Example 8					
	Transpir 0					

50

55

Table 11 (continued)

5	<u></u>							
J	Production	Production o	Production of non-magnetic acicular black					
	Examples	iron-ba	iron-based composite particles					
	and	Addition of	Addition of carbon black fine particles					
10	Production	Edge runne	er treatment	Amount				
,	Comparative			adhered				
!	Examples	Linear load	Time	(calculated				
15		(Kg/cm)	(min)	as C)				
				(wt. %)				
į	Production	40	30	4.76				
	Example 12							
20	Production Example 13	60	20	2.91				
	Production			0 11				
	Example 14	20	30	9.11				
25	Production	35	40	7.00				
	Example 15							
	Production Example 16	<b>4</b> 5	20	4.81				
	Production			4 = 0				
30	Example 17	75	20	4.78				
	Production	60	30	1.97				
	Example 18							
	Production	60	30	3.39				
35	Example 19 Production							
į	Example 20	30	30	8.26				
	Production	40	30	4.30				
40	Example 21	<del>-</del>	50	4.50				
40	Production	60	30	_				
	Comparative							
	Example 6							
	Production	60	30	0.01				
45	Comparative							
	Example 7							
	Production	60	30	2.91				
j	Comparative		ı					
50	Example 8							

55

### Table 12

5					
5	Production	Properties	of non-mag	metic acid	cular black
	Examples		-based comp	osite part	icles
	and	Average	Average	Aspect	Geometrica
	Production	major	minor	ratio	1 standard
10	Comparative	axis	axis	( - )	deviation
	Examples	diameter	diameter		(-)
		(hw)	(µm)		
	Production	0.143	0.0210	6.8:1	1.37
15	Example 12 Production				
	Example 13	0.186	0.0242	7.7:1	1.34
	Production				
	Example 14	0.156	0.0210	7.4:1	1.43
20	Production	0.241	0.0272	8.9:1	1.36
	Example 15	0.241	0.0272	0.9:1	1.36
	Production	0.191	0.0221	8.6:1	1.41
	Example 16		0.022		
25	Production	0.143	0.0211	6.8:1	1.37
	Example 17				
	Production	0.186	0.0242	7.7:1	1.34
	Example 18				
30	Production	0.157	0.0211	7.4:1	1.43
	Example 19 Production				
	Example 20	0.241	0.0272	8.9:1	1.36
	Production				
35	Example 21	0.190	0.0222	8.6:1	1.41
	Production	0.143	0.0211	6.8:1	1.37
	Comparative	0.143	0.0211	0.6:1	1.3/
	Example 6	i			
40	Production	0.240	0.0272	8.8:1	1.37
	Comparative				
	Example 7				
	Production	0.241	0.0273	8.8:1	1.36
45	Comparative				
	Example 8				

50

55

Table 12 (continued)

5		γ				
3	Production	Properti		_	acicular bla	ack iron-
	Examples	<u> </u>	based	composite p	particles	
	and	BET	Mn	Volume	Black-	Carbon
	Production	specifi	content	resisti-	ness	black
10	Comparative	С	(wt. %)	vity	(L*	desorp-
	Examples	surface		( <b>Ω•</b> cm)	value)	tion
		area			(-)	percen-
		(m <sup>2</sup> /g)				tage
15						(%)
	Production	54.8	_	$1.8 \times 10^{4}$	18.9	6.8
	Example 11					
	Production	47.2	_	$7.2 \times 10^{5}$	19.3	8.9
	Example 12					
20	Production	54.3	8.1	$3.2 \times 10^2$	17.6	5.6
	Example 13					
•	Production	89.1	-	$2.6 \times 10^{3}$	19.6	8.3
	Example 14					
25	Production	116.8	6.9	$4.3 \times 10^{3}$	18.1	5.6
	Example 15					
	Production	54.6	-	$2.9 \times 10^{4}$	18.9	4.6
	Example 16					
30	Production	47.3	-	$3.6 \times 10^{5}$	19.7	4.2
	Example 17 Production					
	Example 18	54.1	8.0	$3.1 \times 10^4$	18.1	3.8
	Production			_		
35	Example 19	87.3	-	$4.8 \times 10^{3}$	17.3	3.2
	Production					
	Example 20	113.6	7.1	$5.6 \times 10^4$	18.6	2.9
	Production					
	Comparative	55.0	-	$4.1 \times 10^{7}$	28.5	-
40	Example 6					
	Production	00.5		0.0.107		
	Comparative	88.6	-	$8.2 \times 10^{7}$	34.1	_
	Example 7					
45	Production	00 0		7 6 106	20.2	42.2
	Comparative	89.0		$7.6 \times 10^6$	30.3	43.2
	Example 8					
				لــــــــــــــــــــــــــــــــــــ	<u> </u>	

Table 13

10

15

20

25

30

35

40

45

Production of Properties Examples non-magnetic coating of coating composition and composition Comparative Weight ratio Kind of non-Viscosity Examples magnetic of particles (CP) to resin particles (-)Production 5.0 Example 23 410 Example 11 Production Example 24 5.0 358 Example 12 Production 5.0 Example 25 435 Example 13 Production 5.0 Example 26 410 Example 14 Production 5.0 Example 27 461 Example 15 Production Example 28 5.0 512 Example 16 Production 5.0 333 Example 29 Example 17 Production 5.0 Example 30 281 Example 18 Production 5.0 Example 31 384 Example 19 Production Example 32 5.0 410 Example 20 Production Example 33 5.0 333 Example 21 5.0 Comparative 358 Production Example 27 Comparative Example 6 Comparative Production 5.0 1,024 Example 28 Comparative Example 7 5.0 3,840 Comparative Production Example 29 Comparative Example 8

55

Table 13 (continued)

5	
10	
15	
20	
25	
30	
35	

Examples	Properties of non-magnetic undercoat layer			
and Comparative Examples	Thickness (µm)	Gloss (%)	Surface roughness Ra (nm)	Young's modulus (relative value)
Example 23	3.5	198	6.8	128
Example 24	3.5	194	7.2	123
Example 25	3.4	193	7.3	126
Example 26	3.5	186	8.8	123
Example 27	3.5	191	7.4	127
Example 28	3.4	189	7.8	122
Example 29	3.6	196	6.9	123
Example 30	3.5	198	6.8	125
Example 31	3.5	195	6.9	124
Example 32	3.6	202	6.2	129
Example 33	3.5	206	6.0	124
Comparative Example 27	3.5	179	9.8	120
Comparative Example 28	3.6	173	12.3	118
Comparative Example 29	3.7	153	21.6	116

### Table 13 (continued)

•	_	,	

Examples	Properties of non-magnetic undercoat				
	layer				
and	Linear absorption	Surface resistivity			
Comparative	(µm <sup>-1</sup> )	$(\Omega/ exttt{sq})$			
Examples					
Example 23	1.82	2.1 x 10 <sup>9</sup>			
Example 24	1.59	8.6 x 10 <sup>9</sup>			
Example 25	1.50	4.6 x 10 <sup>9</sup>			
Example 26	2.38	1.8 x 10 <sup>9</sup>			
Example 27	1.81	8.9 x 10 <sup>9</sup>			
Example 28	1.53	2.6 x 10 <sup>9</sup>			
Example 29	1.53	7.6 x 10 <sup>9</sup>			
Example 30	1.46	9.6 x 10 <sup>9</sup>			
Example 31	1.53	6.3 x 10 <sup>9</sup>			
Example 32	2.16	9.2 x 10 <sup>9</sup>			
Example 33	1.51	9.4 × 10 <sup>9</sup>			
Comparative		$6.7 \times 10^{13}$			
Example 27	0.91				
Comparative	0.05	$5.6 \times 10^{12}$			
Example 28	0.97				
Comparative	1 10	$2.0 \times 10^{11}$			
Example 29	1.18	<u> </u>			

### Table 14

5						
5			Production of			
	Examples	magnetic recording medium				
	and	Kind of non-	Kind of	Weight ratio		
	Comparative	magnetic	magnetic	of particles		
10	Examples	undercoat	particles	to resin		
		layer		( - )		
	Example 34	Example 23	Particles used	5.0		
		27.00	in Example 2			
15	Example 35	Example 24	Particles used	5.0		
		270000020	in Example 2			
	Example 36	Example 25	Magnetic	5.0		
		indipic is	particles (1)			
	Example 37	Example 26	Magnetic	5.0		
20		Dramp10 D0	particles (1)			
	Example 38	Example 27	Magnetic	5.0		
			particles (2)			
	Example 39	Example 28	Magnetic	5.0		
25			particles (2)			
	Example 40	Example 29	Particles used	5.0		
		<u> </u>	in Example 2			
	Example 41	Example 30	Magnetic	5.0		
30			particles (1)			
	Example 42	Example 31	Magnetic	5.0		
		-	particles (2)			
	Example 43	Example 32	Magnetic	5.0		
05			particles (3)			
35	Example 44	Example 33	Magnetic	5.0		
			particles (4)			
	Comparative	Comparative	Magnetic	5.0		
	Example 30	Example 27	particles (1)			
40	Comparative	Comparative	Magnetic	5.0		
	Example 31	Example 28	particles (1)			
	Comparative:	Comparative	Magnetic	5.0		
	Example 32	Example 29	particles (1)			

## Table 14 (continued)

5	
10	
15	
20	
25	
30	
35	
40	

	<u> </u>					
Examples	Properties of					
and	magnetic recording medium					
Comparative	Thickness	Coercive	Br/Bm	Gloss		
Examples	of .	force Hc	(-)	(웅)		
	magnetic	(0e)				
	coating film					
	(µm)					
Example 34	1.0	1,971	0.88	229		
Example 35	1.1	1,968	0.87	228		
Example 36	1.0	1,983	0.87	226		
Example 37						
7	1.1	1,975	0.86	210		
Example 38	1.0	1,769	0.87	218		
Example 39	1.1	1,773	0.87	211		
Example 40	1.1	1,976	0.88	228		
Example 41			0.88	223		
Example 42	1.0	1,988	0.00	_223		
Example 42	1.0	1,783	0.89	213		
Example 43	1.1	976	0.90	195		
Example 44	1,0	920	0.90	199		
Comparative						
Example 30	1.0	1,974	0.86	193		
Comparative	1 1	1 074	0.05	101		
Example 31	1.1	1,974	0.85	191		
Comparative Example 32	1.1	1,970	0.84	168		
Example 37		1,7,0	0.01			

### Table 14 (continued)

7		Properties of				
Example:	5	magn	etic re	cording med	ium	
Comparati	ve Surfa	.ce Yo	ung's	Linear	Surface	
Example:	roughn	ess mo	dulus	absorption	resistivity	
	Ra	(re	lative	(µm <sup>-1</sup> )	$(\Omega/\text{sq})$	
	(rim	) va	ılue)			
Example	6.0		127	1.86	5.6 x 10 <sup>8</sup>	
Example :	6.0		126	1.89	$2.6 \times 10^{8}$	
Example	6.2		128	1.87	$3.8 \times 10^{8}$	
Example	7.4		125	2.53	$2.5 \times 10^8$	
Example :	6.9		131	1.71	$3.1 \times 10^9$	
Example :	7.1		126	1.96	8.6 x 10 <sup>8</sup>	
Example			127	1.93	$8.6 \times 10^8$	
Example			130	1.88	$3.2 \times 10^8$	
Example 4	6.8		127	1.94	$2.6 \times 10^8$	
Example 4	7.7		134	2.38	$5.1 \times 10^9$	
Example			128	1.63	$1.3 \times 10^9$	
Comparati Example			123	1.18	8.1 x 10 <sup>10</sup>	
Comparati Example	ve	0 :	121	1.25	$3.6 \times 10^{11}$	
Comparati Example	ve	6	118	1.38	8.8 x 10 <sup>10</sup>	

### Table 15

5	Production		Production o	- 1
	Examples		magnetic acicul	
	and	Kind of core	iron-based co	mposite
	Production	particles	particle	es
40	Comparative			
10	Examples		Addition of m	odified
			polysiloxane	
			Additive	s
			Kind	Amount
15				added
				(part by
				weight)
	Production	Core particles 1	BYK-080	1.0
20	Example 23		D11. 000	1.0
	Production	Core particles 2	BYK-080	1.0
	Example 24		2111 000	2.0
	Production	Core particles 3	BYK-322	2.0
	Example 25			2.0
25	Production	Core particles 4	TSF4446	1.0
	Example 26			
	Production	Core particles 5	TSF4460	3.0
	Example 27			
30	Production	Core particles 6	BYK-080	0.5
	Example 28			
	Production	Core particles 7	BYK-310	1.5
	Example 29			
35	Production	Core particles 8	BYK-322	2.5
	Example 30			
	Production	Core particles 9	TSF4446	1.0
	Example 31			
	Production	Core particles 10	TSF4460	1.0
40	Example 32			
	Production	Core particles 1	BYK-080	1.0
	Comparative			
	Example 9			
45	Production	Core particles 4	BYK-080	0.5
	Comparative	İ		
	Example 10			
	Production	Core particles 4	BYK-080	0.005
50	Comparative			
	Example 11			

## Table 15 (continued)

	Production of non-magnetic acicular black				
Examples	iron-based composite particles				
and	Addition	of modified po	lysiloxane		
Production	Edge runne	r treatment	Amount		
	l and the second		coated		
Comparative					
Examples	Linear load	Time	(calculated		
	(Kg/cm)	(min)	as Si)		
			(wt. %)		
Production	40	30	0.18		
Example 23			J. 20		
Production	45	40	0.17		
Example 24					
Production	60	20	0.35		
Example 25 Production					
Example 26	30	30	0.17		
Production		2.0	2.50		
Example 27	70	30	0.50		
Production	45	20	0.09		
Example 28	40	20	0.09		
Production	30	20	0.25		
Example 29		_ •			
Production	60	30	0.43		
Example 30					
Production Example 31	40	30	0.17		
Production					
Example 32	60	40	0.16		
Production	60	30	0.17		
Comparative	00	30	0.17		
Example 9					
Production	60	30	0.08		
Comparative					
Example 10					
Production	60	30	$9.0 \times 10^{-4}$		
Comparative		l			
Example 11					

## Table 15 (continued)

5						
5	Production	Production of non-magnetic acicular black				
	Examples	iron-based composite particles				
	and	Addition of carbon black fine parti				
10	Production	Carbon black fine particles				
	Comparative	Kind	Amount added			
	Examples		(part by weight)			
	Production	7	9.0			
15	Example 23	Α	<b>9.</b> 0			
	Production	A	5.0			
	Example 24	**	3.0			
	Production	В	7.0			
20	Example 25		7.0			
	Production	В	10.0			
	Example 26					
25	Production	С	10.0			
	Example 27	The second secon				
	Production	A	7.5			
	Example 28 Production					
	Example 29	A	10.0			
30	Production					
	Example 30	В	5.0			
	Production	n	3.0			
	Example 31	В	3.0			
35	Production	С	5.0			
	Example 32		3.0			
	Production	_	_			
	Comparative					
40	Example 9					
	Production	A	0.01			
	Comparative					
	Example 10					
45	Production	В	5.0			
45	Comparative					
	Example 11					

65

50

### Table 15 (continued)

5	Production	Production of non-magnetic acicular black			
Ü	1	_			
	Examples	iron-based composite particles			
	and	Addition of	ine particles		
10	Production	Edge runne	r treatment	Amount	
	Comparative			adhered	
	Examples	Linear load	Time	(calculated	
15		(Kg/cm)	(min)	as C)	
70				(wt. %)	
	Production	60	30	8.35	
	Example 23			<u> </u>	
20	Production	40	30	4.75	
	Example 24 Production			<del>-</del>	
	Example 25	40	20	6.53	
	Production	35	20	9.11	
25	Example 26				
	Production	35	30	9.09	
	Example 27				
	Production Example 28	30	20	6.98	
30	Production	30	20	9.09	
	Example 29	30	20	9.09	
	Production	40	30	4.77	
35	Example 30				
	Production	45	45	2.92	
	Example 31 Production				
	Example 32	60	30	4.73	
40	Production			_	
	Comparative	_	_	_	
	Example 9				
	Production	60	30	0.01	
45	Comparative				
	Example 10				
	Production Comparative	60	30	4.76	
	Example 11		į		
50	2.samp10 11		1		

Table 16

Properties of non-magnetic acicular black

5	

Production

10

15

20

25

30

35

40

45

Examples iron-based composite particles and Average Average Aspect Geometrica Production major minor ratio l standard Comparative axis axis (-)deviation Examples diameter diameter (-)(mm) (mm) Production 0.143 0.0211 6.8:1 1.36 Example 23 Production 0.187 0.0241 7.8:1 1.34 Example 24 Production 0.156 7.4:1 0.0211 1.43 Example 25 Production 0.241 0.0272 8.9:1 1.35 Example 26 Production 0.191 0.0221 8.6:1 1.42 Example 27 Production 0.143 6.7:1 1.36 0.0213 Example 28 Production 0.186 0.0243 7.7:1 1.34 Example 29 Production 0.158 0.0212 7.5:1 1.43 Example 30 Production 0.241 0.0273 8.8:1 1.35 Example 31 Production 0.191 8.6:1 1.41 0.0221 Example 32 Production 0.143 0.0210 6.8:1 1.38 Comparative Example 9 Production 0.240 0.0271 8.9:1 1.38 Comparative Example 10 Production 0.241 0.0273 8.8:1 1.36 Comparative Example 11

50

# Table 16 (continued)

5	Production Examples	Properties of black iron-based composite particles				
	and	BET	Mn	Volume	Black-	Carbon
	Production	specific	content	resisti-	ness	black
	Comparative	surface	(wt. %)	vity	L*	desorp-
10	Examples	area	(WC. 6)	(Ω•cm)	value)	tion
	Drampies	$(m^2/g)$	i	(uz · Cm)	(-)	percen-
		(111.79)			` '	tage
						(%)
15	Production	55.4	_	$2.6 \times 10^{3}$	17.9	5.9
	Example 23					
	Production	46.9	-	$3.2 \times 10^4$	18.9	6.3
	Example 24		_			
20	Production	54.2	8.2	$9.6 \times 10^{3}$	18.6	7.3
	Example 25					
	Production	88.3	-	$5.4 \times 10^{3}$	17.6	6.9
	Example 26					
25	Production Example 27	115.8	6.9	$1.6 \times 10^{3}$	17.3	8.3
	Production					
	Example 28	55.8	_	$1.6 \times 10^4$	18.1	4.1
	Production	15.0	:		4-0	
30	Example 29	47.3	-	$4.1 \times 10^{3}$	17.8	3.6
	Production	54.1	8.0	$4.8 \times 10^{4}$	18.6	4.8
	Example 30	24.1	0.0	4.6 X 10-	10.0	4.0
	Production	86.3	_	$1.0 \times 10^{5}$	19.5	3.2
<i>35</i>	Example 31	00.5		1.0 X 10	17.5	J.2
	Production	112.8	7.1	$3.6 \times 10^4$	18.6	2.6
	Example 32					
	Production	54.6	_	$6.1 \times 10^{7}$	27.3	_
40	Comparative					
40	Example 9					
1	Production	87.9	_	$7.6 \times 10^7$	32.9	-
	Comparative					
	Example 10					
45	Production	88.9	_	$8.1 \times 10^{6}$	30.1	51.3
	Comparative					
	Example 11					

68

50

### Table 17

5	

Production of

Properties

1	rroddetron or		rropercies	
Examples	non-magnetic coating		of coating composition	
and		composition		
Comparativ	Kind of non-	Weight ratio	Viscosity	
e Examples	magnetic	of particles	(cP)	
	particles	to resin		
		(-)		
Example 45	Production	5.0	384	
	Example 22			
Example 46	Production	5.0	410	
	Example 23	3.0		
Example 47	Production	5.0	333	
Drampre 17	Example 24	J. 0	<i></i>	
Example 48	Production	5.0	435	
DAGINDIC 40	Example 25	3.0	133	
Example 49	Production	5.0	461	
Drampic 43	Example 26	J. 0	40T	
Example 50	Production	5.0	358	
Example 30	Example 27	3.0	330	
Example 51	Production	5.0	310	
DXMIDIC JI	Example 28	3.0	310	
Example 52	Production	5.0	410	
LXMmpre JZ	Example 29		410	
Example 53	Production	5.0	384	
DXMIDIC 33	Example 30	3.0	204	
Example 54	Production	5.0	333	
DXMIDIC 34	Example 31	] 3.0	333	
Example 55	Production	5.0	310	
Example 33	Example 32	3.0	310	
Comparativ	Production	5.0	410	
e Example	Comparative			
33	Example 9			
Comparativ	Production	5.0	1,280	
e Example	Comparative			
34	Example 10			
Comparativ	Production	5.0	3,584	
e Example	Comparative	1	,	
35	Example 11			
		<u></u>		

## Table 17 (continued)

5					
5	Examples	Properties of non-magnetic undercoat layer			
10	and Comparative Examples	Thickness (µm)	Gloss (%)	Surface roughness Ra (nm)	Young's modulus (relative value)
	Example 45	3.5	196	6.8	128
15	Example 46	3.5	191	7.1	123
	Example 47	3.4	193	6.9	125
20	Example 48	3.5	190	7.2	123
	Example 49	3.5	186	7.8	127
25	Example 50	3.5	186	7.9	123
	Example 51	3.6	196	6.8	123
	Example 52	3.5	196	6.8	125
30	Example 53	3.5	203	6.3	125
	Example 54	3.6	205	6.3	129
35	Example 55	3.5	198	6.8	125
	Comparative Example 33	3.5	176	10.0	121
	Comparative Example 34	3.7	173	14.4	117
40	Comparative Example 35	3.7	151	25.6	113

### Table 17 (continued)

5		
10		
15		
20		
25		
30		
35		

Examples	Properties of non-magnetic undercoat				
	layer				
and	Linear absorption	Surface resistivity			
Comparative	$(\mu m^{-1})$	$(\Omega/\mathrm{sq})$			
Examples	,				
Example 45		$1.8 \times 10^9$			
DAGRETC 45	1.84				
Example 46	0.00	$3.4 \times 10^9$			
	2.20				
Example 47	1 61	$5.1 \times 10^9$			
	1.61	0 1 100			
Example 48	1.76	$8.1 \times 10^9$			
·	1.70	$1.0 \times 10^{9}$			
Example 49	2.31	1.0 x 10			
		$1.1 \times 10^9$			
Example 50	2.33				
Example 51		$3.1 \times 10^9$			
Example 21	1.86				
Example 52		$1.2 \times 10^9$			
HAMIPTE 32	2.30				
Example 53		$6.8 \times 10^{9}$			
	1.58				
Example 54	1 42	$2.1 \times 10^{10}$			
	1.43	4.6 x 10 <sup>9</sup>			
Example 55	1.57	4.6 X 10 <sup>3</sup>			
Comparative	1.57	6.3 x 10 <sup>13</sup>			
Example 33	0.93	0.3 X 10=-			
Comparative		$8.7 \times 10^{12}$			
_	1.01	0./ X 10-2			
Example 34	1.01	1 6 2 1011			
Comparative	1.19	$1.6 \times 10^{11}$			
Example 35	1.19	<u> </u>			

# Table 18

_						
5		Production of				
	Examples	magnetic recording medium				
	and	Kind of non-	Kind of	Weight ratio		
	Comparative	magnetic	magnetic	of particles		
10	Examples	undercoat	particles	to resin		
		layer		(-)		
	Example 56	Example 45	Particles used in Example 2	5.0		
15	Example 57	Example 46	Particles used in Example 2	5.0		
	Example 58	Example 47	Magnetic particles (1)	5.0		
20	Example 59	Example 48	Magnetic particles (1)	5.0		
	Example 60	Example 49	Magnetic particles (2)	5.0		
25	Example 61	Example 50	Magnetic particles (2)	5.0		
	Example 62	Example 51	Particles used in Example 2	5.0		
30	Example 63	Example 52	Magnetic particles (1)	5.0		
30	Example 64	Example 53	Magnetic particles (2)	5.0		
	Example 65	Example 54	Magnetic particles (3)	5.0		
35	Example 66	Example 55	Magnetic particles (4)	5.0		
	Comparative	Comparative	Magnetic	5.0		
	Example 36	Example 33	particles (1)			
40	Comparative	Comparative	Magnetic	5.0		
	Example 37	Example 34	particles (1)			
	Comparative	Comparative	Magnetic	5.0		
	Example 38	Example 35	particles (1)			

# Table 18 (continued)

5	
10	
15	
20	
25	
30	
35	
40	

Essembles	Properties of					
Examples and	magnetic recording medium					
Comparative	Thickness	Coercive	Br/Bm	Gloss		
Examples	of	force Hc	(-)	(%)		
	magnetic	(0e)				
	layer					
	(µm)					
Example 56	1.0	1,979	0.88	226		
Example 57	1.1	1,960	0.87	223		
Example 58	1.0	1,980	0.87	223		
Example 59	1.0	1,973	0.87	215		
Example 60	1.1	1,769	0.87	210		
Example 61	1.1	1,768	0.88	212		
Example 62	1.0	1,983	0.88	228		
Example 63	1.1	1,989	0.87	227		
Example 64	1.0	1,783	0.89	218		
Example 65	1.0	972	0.89	190		
Example 66	1,0	915	0.90	190		
Comparative Example 36	1.0	1,971	0.86	189		
Comparative Example 37	1.0	1,972	0.85	181		
Comparative Example 38	1.1	1,971	0.83	159		

# Table 18 (continued)

5	Examples and	Properties of magnetic recording medium				
10	Comparative Examples	Surface roughness	Young's modulus	Linear absorption	Surface resistivity	
		Ra (nm)	(relative value)	(µm <sup>-1</sup> )	$(p_{ extsf{R}}/\Omega)$	
15	Example 56	6.2	128	1.87	4.8 x 108	
	Example 57	6.0	124	2.26	$4.0 \times 10^{8}$	
20	Example 58	6.0	127	1.68	8.2 x 10 <sup>8</sup>	
	Example 59	6.4	124	2.01	6.0 x 108	
	Example 60	6 <b>.6</b>	129	2.46	2.3 x 10 <sup>9</sup>	
25	Example 61	6.4	125	2.48	$2.1 \times 10^{8}$	
	Example 62	6.2	125	1.96	5.1 x 10 <sup>8</sup>	
30	Example 63	6.2	130	2.61	$1.2 \times 10^8$	
	Example 64	6.6	126	1.63	$7.3 \times 10^8$	
35	Example 65	6.8	134	1.46	1.6 x 10 <sup>9</sup>	
	Example 66	7.0	126	1.66	$8.1 \times 10^9$	
	Comparative Example 36	8.2	121	1.20	$9.6 \times 10^{10}$	
40	Comparative Example 37	10.1	121	1.26	5.3 x 10 <sup>11</sup>	
	Comparative Example 38	17.6	118	1.37	$8.2 \times 10^{10}$	

### Table 19

5	Danadustian		D		
J	Production		Production o	-	
	Examples		magnetic acicul		
	and	Kind of core	iron-based co	- 1	
	Production	particles	particle		
10	Comparative		Addition of te		
	Examples		modified polys	modified polysiloxane	
			Additive	es	
		·	Kind	Amount	
45				added	
15				(part by	
				weight)	
	Production	Core particles 1	TSF4770	2.0	
	Example 34	core pareferes i	151 4770	2.0	
20	Production	Core particles 2	TSF4751	0.5	
	Example 35	Core particles 2	1014/01	0.5	
	Production	Core particles 3	XF3905	5.0	
	Example 36	core particles 5	AF 3 9 0 3	3.0	
25	Production	Core particles 4	YF3804	2.0	
	Example 37	Core partities 4	11,2004	2.0	
	Production	Core particles 5	TSF4770	1.0	
	Example 38	core parcicles 3	1514//0	1.0	
	Production	Core particles 6	TSF4770	1.0	
30	Example 39	core particles o	1554770	1.0	
	Production	Core particles 7	TSF4751	0.5	
	Example 40	core partities /	1214/21	0.5	
	Production	Core particles 8	XF3905	2.0	
35	Example 41	core parcicles o	AF 3903	2.0	
	Production	Core particles 9	YF3804	1.5	
	Example 42	core particles 3	11.2004	1.7	
	Production	Core particles 10	TSF4770	3.0	
40	Example 43	core particles io	1314//0	3.0	
40	Production	Core particles 1	TSF4770	1.0	
	Comparative	core particles i	1214//0	1.0	
	Example 12				
	Production	Core particles 4	TSF4770	1 0	
45	Comparative	core particles 4	1214//0	1.0	
	Example 13				
	Production	Coro portigles 4	mc=/220	0.005	
	Comparative	Core particles 4	, TSF4770	0.005	
50	Example 14				
				J	

### Table 19 (continued)

5							
		Production of	f non-magnetic a	acicular black			
	Examples	iron-ba	sed composite p	articles			
	and	Addition of terminal-modified polysiloxane					
10	Comparative	Edge runne	Amount				
	Examples		coated				
	_	Linear load	Time	(calculated			
15				-			
		(Kg/cm)	(min)	as Si)			
	Production			(wt. %)			
	Example 34	30	30	0.45			
20	Production	60	20	0.12			
	Example 35		20	0.12			
	Production Example 36	40	40	1.10			
25	Production						
25	Example 37	30	40	0.44			
	Production	50	30	0.23			
	Example 38		30	0.23			
30	Production	70	20	0.21			
	Example 39 Production						
	Example 40	60	30	0.13			
	Production	55	30	0.46			
35	Example 41			0.10			
	Production	35	30	0.36			
	Example 42 Production						
40	Example 43	45	20	0.65			
	Production	60	30	0.25			
	Comparative	. 50		0.25			
	Example 12						
45	Production Comparative	60	30	0.25			
	Example 13						
;	Production	60	30	1.3 × 10 <sup>-3</sup>			
	Comparative	00	۲۰	1.7 × 10 -			
50	Example 14						

# Table 19 (continued)

5	Production Production of non-magnetic acicul				
	Examples	black iron-based	composite particles		
	and	Addition of carbon black fine particles			
10	Production	Carbon black fine particles			
	Comparative	Kind	Amount added		
	Examples		(part by weight)		
45	Production	A	11.0		
20	Example 34		11.0		
	Production Example 35	A	5.0		
	Production Example 36	В	7.5		
	Production Example 37	В	10.0		
25	Production	С	15.0		
	Example 38		13.0		
	Production Example 39	A	10.0		
	Production	A	10.0		
30	Example 40				
30	Production Example 41	В	7.5		
	Production		10.0		
	Example 42	В	12.0		
35	Production	С	15.0		
	Example 43				
	Production	-	-		
	Comparative Example 12				
40	Production				
	Comparative	A	0.01		
	Example 13				
	Production	В	5.0		
45	Comparative	5	3.0		
	Example 14				

77

50

# Table 19 (continued)

5	Production	Production o	Production of non-magnetic acicular black				
	Examples	iron-based composite particles					
	and	Addition of carbon black fine particles					
10	Production		er treatment	Amount			
,,	Comparative	Lage Lamie	or creatment	adhered			
	1	- ' 1 1					
	Examples	Linear load	Time	(calculated			
15		(Kg/cm)	(min)	as C)			
				(wt. %)			
	Production	30	30	9.92			
	Example 34 Production						
20	Example 35	45	60	4.77			
	Production	40	<b>3</b> E	C 00			
	Example 36	40	35	6.98			
0.5	Production	55	60	9.10			
25	Example 37						
	Production	50	40	13.21			
	Example 38 Production						
30	Example 39	70	50	9.08			
	Production	60	45	9.10			
	Example 40	00		J.10			
	Production	30	40	7.03			
35	Example 41						
	Production Example 42	35	50	10.71			
	Production	4.0	3.0	12.46			
	Example 43	40	30	13.46			
40	Production	_	-	-			
	Comparative						
	Example 12						
	Production Comparative	60	30	0.01			
45	Example 13		:				
	Production	60	20	4.71			
	Comparative	ا	30	4./1			
50	Example 14		<u> </u>				
<del>-</del>							

55

Table 20

Properties of non-magnetic acicular black

iron-based composite particles

Production Examples

10

15

20

25

30

35

40

45

and Average Average Aspect Geometrical Production major minor ratio standard Comparative axis (-)deviation axis Examples diameter diameter (-)(µm) (µm) Production 0.143 0.0212 6.7:1 1.36 Example 34 Production 0.186 0.0242 7.7:1 1.34 Example 35 Production 0.158 0.0212 7.5:1 1.43 Example 36 Production 0.240 0.0273 8.8:1 1.35 Example 37 Production 0.191 0.0221 8.6:1 1.42 Example 38 Production 0.0212 6.8:1 1.37 0.144 Example 39 Production 0.186 0.0242 7.7:1 1.35 Example 40 Production 0.159 0.0212 7.5:1 1.43 Example 41 Production 8.8:1 1.36 0.241 0.0273 Example 42 Production 0.190 0.0222 8.6:1 1.41 Example 43 Production 6.8:1 0.143 0.0210 1.38 Comparative Example 12 Production 0.240 0.0272 8.8:1 1.38 Comparative Example 13 Production 0.240 0.0272 8.8:1 1.37 Comparative Example 14

50

Table 20 (continued)

5	Production	Properties of non-magnetic acicular black				
	Examples	-	iron-base	d composite		
	and	BET	Mn	Volume	Black-	Carbon
	Production	specific		resisti-	ness	black
10	Comparative	surface	(wt. %)	vity	(上*	desorp-
	Examples	area		$(\Omega ullet \mathtt{cm})$	value)	tion
		$(m^2/g)$			( – )	percen-
						tage
15						(%)
	Production	55.8	-	$2.1 \times 10^{3}$	17.3	8.8
	Example 34					
	Production	47.1	-	$3.8 \times 10^4$	18.6	6.1
	Example 35					
20	Production	55.1	8.2	$9.6 \times 10^{3}$	17.6	7.3
	Example 36					
	Production Example 37	89.3	-	$4.1 \times 10^{3}$	17.3	8.3
	Production		-	2		
25	Example 38	116.0	6.8	$7.2 \times 10^2$	16.9	9.6
	Production					
	Example 39	55.6	-	$6.1 \times 10^3$	17.3	4.6
	Production	40.5		T 1 103	15.5	4 6
30	Example 40	48.3	_	$7.1 \times 10^3$	17.5	4.6
	Production	53.8	7.9	$6.9 \times 10^{4}$	17.9	3.6
	Example 41	53.8	7.9	0.9 X 10-	17.9	3.0
	Production	88.8	_	$3.9 \times 10^{3}$	17.1	3.8
35	Example 42	00.0		3.9 X 10	47.4	3.0
	Production	113.6	7.0	$2.9 \times 10^{3}$	16.5	4.3
	Example 43	113.0	,	2., 1. 20	20.5	
	Production	55.1	_	$5.1 \times 10^{7}$	28.6	_
40	Comparative	33,1				
	Example 12					
	Production	88.2	_	$8.6 \times 10^{7}$	33.2	_
	Comparative					
	Example 13					
45	Production	89.6	-	$8.8 \times 10^{6}$	29.5	48.3
	Comparative	·				-
	Example 14					

50

# Table 21

5	
10	
15	
20	
25	
30	
35	
40	

5

	Produc	tion of	Properties
Examples	non-magnetic coating		of coating
and	composition		1
			composition
Comparative	Kind of non-	Weight ratio	Viscosity
Examples	magnetic	of particles	(cP)
	particles	to resin	
		(-)	
Example 67	Production	5.0	310
	Example 33		
Example 68	Production	5.0	358
	Example 34		
Example 69	Production	5.0	333
Drampre 03	Example 35	3.0	223
Example 70	Production	5.0	410
Example /0	Example 36	5.0	410
B1- 71	Production	۲ ۸	435
Example 71	Example 37	5.0	435
D1- 70	Production	5.0	222
Example 72	Example 38	5.0	333
	Production	F 0	204
Example 73	Example 39	5.0	384
71- 74	Production	г о	410
Example 74	Example 40	5.0	410
_ ,	Production		210
Example 75	Example 41	5.0	310
7 7 7	Production	- ^	4.1.0
Example 76	Example 42	5.0	410
	Production	- ^	
Example 77	Example 43	5.0	333
Comparative	Production	5.0	435
Example 39	Comparative	3.0	
	Example 12		
Comparative	Production	5.0	1,254
_	Comparative	٦.٥	1,434
Example 40	1 -		
Composition	Example 13	E ^	4 000
Comparative	Production	5.0	4,096
Example 41	Comparative		
	Example 14		
		1	

# Table 21 (continued)

5	Examples	Properties of non-magnetic undercoat layer			
10	and Comparative Examples	Thickness (µm)	Gloss (%)	Surface roughness Ra (nm)	Young's modulus (relative value)
	Example 67	3.5	196	6.5	128
15	Example 68	3.5	193	6.9	123
	Example 69	3.4	196	6.7	125
20	Example 70	3.5	191	7.3	124
20	Example 71	3.5	190	7.4	128
	Example 72	3.5	193	6.8	123
25	Example 73	3.6	196	6.6	124
	Example 74	3.5	198	6.5	126
30	Example 75	3.4	201	6.3	125
	Example 76	3.5	205	6.0	128
	Example 77	3.6	198	6.3	125
35	Comparative Example 39	3.5	180	10.8	121
	Comparative Example 40	3.5	176	14.0	116
40	Comparative Example 41	3.5	153	21.6	111

# Table 21 (continued)

5	

Examples	Properties of non-magnetic undercoat layer				
and Comparative Examples	Linear absorption (µm <sup>-1</sup> )				
Example 67	1.87	2.3 x 10 <sup>9</sup>			
Example 68	2.48	2.6 x 10 <sup>9</sup>			
Example 69	1.65	6.8 x 10 <sup>9</sup>			
Example 70	1.93	2.2 x 10 <sup>9</sup>			
Example 71	2.26	1.6 x 10 <sup>9</sup>			
Example 72	2.66	8.1 × 10 <sup>8</sup>			
Example 73	2.38	1.3 x 10 <sup>9</sup>			
Example 74	2.41	9.8 × 10 <sup>8</sup>			
Example 75	2.29	5.2 x 10 <sup>9</sup>			
Example 76	2.46	8.6 x 10 <sup>8</sup>			
Example 77	2.68	2.6 x 10 <sup>8</sup>			
Comparative Example 39	0.91	$8.7 \times 10^{13}$			
Comparative Example 40	0.96	8.8 x 10 <sup>12</sup>			
Comparative Example 41	1.17	3.1 x 10 <sup>11</sup>			

### Table 22

_				
5			Production of	
	Examples		netic recording	
	and	Kind of non-	Kind of	Weight ratio
	Comparative	magnetic	magnetic	of particles
10	Examples	undercoat	particles	to resin
		layer		( – )
	Example 78	Example 67	Particles used	5.0
		Example 07	in Example 2	
15	Example 79	Example 68	Particles used	5.0
75		Example 00	in Example 2	
	Example 80	Example 69	Magnetic	5.0
			particles (1)	
	Example 81	Example 70	Magnetic	5.0
20		Extemp 20 70	particles (1)	
	Example 82	Example 71	Magnetic	5.0
		2ndiipic /i	particles (2)	•
	Example 83	Example 72	Magnetic	5.0
25		27600.1920 72	particles (2)	
	Example 84	Example 73	Particles used	5.0
		Example 75	in Example 2	
	Example 85	Example 74	Magnetic	5.0
30		210011920 / 1	particles (1)	
30	Example 86	Example 75	Magnetic	5.0
			particles (2)	
	Example 87	Example 76	Magnetic	5.0
		21.00	particles (3)	
35	Example 88	Example 77	Magnetic	5.0
		in in the second	particles (4)	
	Comparative	Comparative	Magnetic	5.0
	Example 42	Example 39	particles (1)	
40	Comparative	Comparative	Magnetic	5.0
	Example 43	Example 40	particles (1)	
	Comparative	Comparative	Magnetic	5.0
	Example 44	Example 41	particles (1)	

### Table 22 (continued)

5	
10	
15	
20	
25	
30	
35	
40	

Examples	Properties of					
and	magnetic recording medium					
Comparative	Thickness	Coercive	Br/Bm	Gloss		
Examples	of	force Hc	(-)	(%)		
	magnetic	(0e)				
	coating					
	film					
	(mu)					
Example 78	1.0	1,978	0.88	226		
Example 79	1.1	1,968	0.87	227		
Example 80	1.1	1,981	0.88	230		
Example 81	1.1	1,986	0.87	218		
Example 82	1.0	1,773	0.87	218		
Example 83	1.1	1,768	0.87	210		
Example 84	1.0	1,983	0.87	213		
Example 85	1.1	1,983	0.88	213		
Example 86	1.1	1,782	0.88	216		
Example 87	1.1	973	0.90	193		
Example 88	1,1	915	0.90	190		
Comparative Example 42	1.0	1,977	0.86	188		
Comparative Example 43	1.1	1,972	0.86	186		
Comparative Example 44	1.2	1,970	0.84	161		

### Table 22 (continued)

5							
·	Examples		Prope	rties of	İ		
	and	magnetic recording medium					
	Comparative	Surface	Young's	Linear	Surface		
10	Examples	roughness	modulus	absorption	resistivity		
		Ra	(relative	(µm <sup>-1</sup> )	$(\Omega/\mathrm{sq})$		
		(nm)	value)				
15	Example 78	6.0	127	1.86	$3.6 \times 10^8$		
	Example 79	6.3	125	2.43	$2.6 \times 10^{8}$		
20	Example 80	6.0	127	1.68	$2.3 \times 10^9$		
	Example 81	6.4	125	1.73	8.8 x 10 <sup>8</sup>		
	Example 82	6.4	129	2.26	6.4 x 10 <sup>8</sup>		
25	Example 83	6.8	127	2.66	1.6 x 10 <sup>8</sup>		
	Example 84	7.0	127	2.22	$7.1 \times 10^{8}$		
30	Example 85	6.6	130	2.23	$8.8 \times 10^{8}$		
	Example 86	6.6	127	2.05	$2.3 \times 10^9$		
0.5	Example 87	6.2	135	2.38	5.6 × 10 <sup>8</sup>		
35	Example 88	6.4	126	2.41	1.3 x 10 <sup>8</sup>		
	Comparative Example 42	8.1	126	1.19	$7.3 \times 10^{10}$		
40	Comparative Example 43	9.6	121	1.26	6.8 x 10 <sup>11</sup>		
	Comparative Example 44	13.8	118	1.38	8.8 x 10 <sup>10</sup>		

### Table 23

5	Production Examples and Production Comparative	Kind of core particles	Production of non- magnetic acicular bla iron-based composite particles	
10	Examples		Addition	
			fluoroalkyls	
			Additive	
15			Kind	Amount added (part by weight)
20	Production Example 45	Core particles 1	TSL8527	1.0
	Production Example 46	Core particles 2	TSL8233	2.0
25	Production Example 47	Core particles 3	TSL8262	3.0
20	Production Example 48	Core particles 4	TSL8527	4.0
	Production Example 49	Core particles 5	TSL8233	5.0
30	Production Example 50	Core particles 6	TSL8527	2.0
	Production Example 51	Core particles 7	TSL8233	1.5
35	Production Example 52	Core particles 8	TSL8262	2.5
	Production Example 53	Core particles 9	TSL8527	5.5
40	Production Example 54	Core particles 10	TSL8233	1.0
	Production Comparative	Core particles 1	TSL8527	2.0
	Example 15			
<b>4</b> 5	Production Comparative	Core particles 4	TSL8527	3.0
	Example 16			
50	Production Comparative	Core particles 4	TSL8527	0.005
	Example 17			

# Table 23 (continued)

5	Production	Production of non-magnetic acicular black					
	Examples		-				
		iron-based composite particles Addition of fluoroalkylsilane					
	and	Addici		yisiiane			
10	Production	Edge runne	r treatment	Amount			
	Comparative			coated			
	Examples	Linear load	Time	(calculated			
15		(Kg/cm)	(min)	as Si)			
,-				(wt. %)			
	Production	30	30	0.07			
	Example 45	30	30	0.07			
20	Production	60	20	0.11			
	Example 46						
	Production	40	40	0.47			
	Example 47 Production						
25	Example 48	55	20	0.26			
	Production						
	Example 49	40	60	0.26			
	Production	50	30	0.13			
30	Example 50	30	30	0.13			
	Production	30	40	0.08			
	Example 51			0.00			
	Production	35	40	0.39			
35	Example 52						
	Production	60	30	0.36			
	Example 53 Production						
	Example 54	40	20	0.06			
40	Production	60	30	0.13			
	Comparative	00	30	0.13			
	Example 15						
	Production	60	30	0.20			
45	Comparative	_					
	Example 16						
	Production	60	30	$3.3 \times 10^{-4}$			
	Comparative		'				
50	Example 17						

55

# Table 23 (continued)

5	Production	Production of non-ma	agnetic acicular black
	Examples	iron-based com	posite particles
	and	Addition of carbon	black fine particles
10	Production	Carbon black	fine particles
	Comparative	Kind	Amount added
	Examples		(part by weight)
15	Production Example 45	A	9.0
	Production	A	6.0
	Example 46 Production	-	
20	Example 47	В	5.0
	Production Example 48	В	15.0
	Production Example 49	С	15.0
25	Production Example 50	A	10.0
	Production Example 51	A	15.0
30	Production Example 52	В	10.0
	Production Example 53	В	8.0
35	Production Example 54	С	3.0
40	Production Comparative Example 15	-	-
40	Production Comparative Example 16	A	0.01
45	Production Comparative Example 17	В	5.0

89

50

# Table 23 (continued)

Production	Production of non-magnetic acicular black			
Examples	iron-based composite particles			
and	Addition of	carbon black f	ine particles	
Production		er treatment	Amount	
Comparative	_030 _000		adhered	
_			1	
Examples	Linear load	Time	(calculated	
	(Kg/cm)	(min)	as C)	
			(wt. %)	
Production	40	40	8.26	
Example 45		10	0.20	
Production	65	60	5.66	
Example 46				
Production	30	30	4.76	
Example 47 Production	·	50 50		
Example 48	50	13.04		
Production		2.5	12.04	
Example 49	55	35	13.04	
Production	40	20	9.10	
Example 50	40	20	7.10	
Production	60	30	13.07	
Example 51				
Production	45	60	9.11	
Example 52 Production	- 1. 1-1. 1			
Example 53	60	30	7.41	
Production	25	0.5	0 00	
Example 54	35	25	2.92	
Production	_		_	
Comparative			-	
Example 15				
Production	60	30	0.01	
Comparative	-		- <del>-</del>	
Example 16				
Production	60	30	4.77	
Comparative		<b>'</b>		
Example 17				

### Table 24

5					
3	Production				icular black
	Examples		n-based com		
	and	Average	Average	Aspect	Geometrical
	Production	major	minor	ratio	standard
10	Comparative	axis	axis	( – )	deviation
	Examples	diameter	diameter		(-)
		(mm)	(µm)		
	Production	0.144	0.0211	6.8:1	1.37
15	Example 45 Production				
	Example 46	0.187	0.0241	7.8:1	1.35
	Production				
	Example 47	0.158	0.0212	7.5:1	1.43
20	Production	0.014	0.0050		
	Example 48	0.241	0.0273	8.8:1	1.35
	Production	0.190	0.0221	8.6:1	1.42
	Example 49	0.190	0.0221	0.0:1	1.42
25	Production	0.143	0.0212	6.7:1	1.37
	Example 50	0.143	0.0212	0.7.1	4.57
	Production	0.187	0.0243	7.7:1	1.34
	Example 51			·	
30	Production	0.158	0.0211	7.5:1	1.43
	Example 52				
	Production	0.241	0.0273	8.8:1	1.36
	Example 53 Production				
35	Example 54	0.190	0.0221	8.6:1	1.42
	Production				
	Comparative	0.143	0.0210	6.8:1	1.38
	Example 15				
40	Production	0 040	0 0070	8.8:1	1 35
	Comparative	0.240	0.0273	8.8:1	1.37
	Example 16				
	Production	0.240	0.0273	8.8:1	1.37
45	Comparative	0.240	0.02/3	0.0:1	1.3/
45	Example 17				

91

50

# Table 24 (continued)

5	Production	Properties of non-magnetic acicular black iron-					
	Examples		based o	composite pa	articles		
	and	BET	Mn	Volume	Black-	Carbon	
	Production	specific	content	resisti-	ness	black	
10	Comarative.	surface	(wt. %)	vity	(L*	desorp-	
	Examples	area		(Ω•cm)	value)	tion	
		(m <sup>2</sup> /g)			( – )	percen-	
						tage	
15						(%)	
,0	Production	56.1	<b>-</b> .	$9.6 \times 10^{3}$	17.8	8.0	
	Example 45						
	Production	47.3	-	$2.1 \times 10^4$	18.2	6.0	
	Example 46						
20	Production	55.8	8.0	$3.8 \times 10^4$	18.5	7.3	
	Example 47						
	Production	88.9	_	$4.6 \times 10^{3}$	17.1	8.6	
	Example 48 Production						
25		116.3	6.7	$4.1 \times 10^{3}$	16.9	9.3	
	Example 49 Production						
	Example 50	56.6	-	$1.6 \times 10^{4}$	17.3	4.8	
	Production						
30	Example 51	50.3	_	$2.1 \times 10^{3}$	16.7	4.3	
	Production						
	Example 52	54.6	7.9	$9.6 \times 10^{3}$	17.4	3.2	
	Production			1 0 101	45.0		
35	Example 53	88.8	-	$1.9 \times 10^4$	17.9	4.6	
00	Production	115 6	7 ^	0.0 . 104	10 6	1.0	
	Example 54	115.6	7.0	$8.9 \times 10^4$	18.6	1.8	
	Production	56.3		5 0 107	20 5		
	Comparative	56.3	-	$5.8 \times 10^7$	28.5	-	
40	Example 15		:				
	Production	00 3		0 1 107	22.2		
	Comparative	89.3	_	$9.1 \times 10^7$	33.2	_	
	Example 16						
45	Production	90.9	_	$8.6 \times 10^{6}$	29.7	47.6	
	Comparative	30.3	_	0.0 X 10	43.1	4'.0	
	Example 17			'			

92

55

### Table 25

5	
10	
15	
20	
25	
30	
35	
40	

Examples and	Produc non-magne compo	Properties of coating composition	
Comparative Examples	Kind of non- magnetic particles	Weight ratio of particles to resin (-)	Viscosity (cP)
Example 89	Production Example 44	5.0	333
Example 90	Production Example 45	5.0	384
Example 91	Production Example 46	5.0	333
Example 92	Production Example 47	5.0	410
Example 93	Production Example 48	5.0	640
Example 94	Production Example 49	5.0	568
Example 95	Production Example 50	5.0	333
Example 96	Production Example 51	5.0	358
Example 97	Production Example 52	5.0	435
Example 98	Production Example 53	5.0	307
Example 99	Production Example 54	5.0	256
Comparative Example 45	Production Comparative Example 15	5.0	568
Comparative Example 46	Production Comparative Example 16	5.0	1,280
Comparative Example 47	Production Comparative Example 17	5.0	3,072

### Table 25 (continued)

5					
3	Examples	Properties of non-magnetic undercoat			
		layer			
	and	Thickness	Gloss	Surface	Young's
	Comparative	(mm)	(%)	roughness	modulus
10	Examples			Ra	(relative
				(nm)	value)
	Example 89	3.5	195	6.4	127
15	Example 90	3.4	191	6.6	123
	Example 91	3.5	192	6.4	124
20	Example 92	3.5	190	6.8	125
	Example 93	3.5	190	7.0	129
	Example 94	3.5	195	6.4	123
25	Example 95	3.4	198	6.2	123
	Example 96	3.5	199	6.2	126
30	Example 97	3.5	196	6.4	123
	Example 98	3.4	206	6.0	130
	Example 99	3.3	213	5.8	125
35	Comparative	, <u> </u>	150	11 0	400
	Example 45	3.5	178	11.0	120
	Comparative	,	172	12.2	125
	Example 46	3.6	173	13.2	115
40	Comparative	, ,	156	20.0	110
	Example 47	3.7	156	20.8	110

Linear absorption

 $(\mu m^{-1})$ 

#### Table 25 (continued)

Properties of non-magnetic undercoat

layer

Surface resistivity

 $(\Omega/\text{sq})$ 

Examples

and Comparative

Examples

Example 47

10

15

20

25

30

35

40

45

50

 $2.3 \times 10^9$ Example 89 1.84  $2.3 \times 10^9$ Example 90 2.31  $5.1 \times 10^9$ Example 91 1.70  $6.7 \times 10^9$ Example 92 1.63  $3.6 \times 10^{8}$ Example 93 2.64  $3.1 \times 10^{8}$ Example 94 2.66  $3.8 \times 10^9$ Example 95 2.18  $5.0 \times 10^{8}$ Example 96 2.68  $5.6 \times 10^9$ Example 97 2.20  $1.8 \times 10^{9}$ Example 98 2.01  $9.6 \times 10^9$ Example 99 1.53  $7.1 \times 10^{13}$ Comparative 0.90 Example 45  $9.4 \times 10^{12}$ Comparative 0.94 Example 46  $2.3 \times 10^{11}$ Comparative

1.18

### Table 26

5						
3		Production of				
	Examples	magnetic recording medium				
	and	Kind of non-	Kind of	Weight ratio		
	Comparative	magnetic	magnetic	of particles		
10	Examples	undercoat	particles	to resin		
		layer		( - )		
	Example 100	Example 89	Particles used	5.0		
		2.00	in Example 2			
15	Example 101	Example 90	Particles used	5.0		
		27.00.09.20 90	in Example 2			
	Example 102	Example 91	Magnetic	5.0		
		Drumpic 71	particles (1)			
	Example 103	Example 92	Magnetic	5.0		
20			particles (1)			
	Example 104	Example 93	Magnetic	5.0		
		27104.1520 33	particles (2)			
	Example 105	Example 94	Magnetic	5.0		
25			particles (2)			
	Example 106	Example 95	Particles used	5.0		
,			in Example 2			
	Example 107	Example 96	Magnetic	5.0		
30			particles (1)			
	Example 108	Example 97	Magnetic	5.0		
			particles (2)			
:	Example 109	Example 98	Magnetic	5.0		
			particles (3)			
35	Example 110	Example 99	Magnetic	5.0		
l			particles (4)			
	Comparative	Comparative	Magnetic	5.0		
	Example 48	Example 45	particles (1)			
40	Comparative	Comparative	Magnetic	5.0		
	Example 49	Example 46	particles (1)			
	Comparative	Comparative	Magnetic	5.0		
	Example 50	Example 47	particles (1)			

### Table 26 (continued)

5	
10	
15	
20	
25	
30	
35	
40	

	1				
Examples	Properties of				
and	magnetic recording medium				
Comparative	Thickness	Coercive	Br/Bm	Gloss	
Examples	of	force Hc	(-)	(%)	
	magnetic	(0e)			
	coating				
	film				
	(µm)				
Example 100	1.0	1,973	0.88	228	
Example 101	1.1	1,968	0.87	220	
Example 102	1.0	1,981	0.87	225	
Example 103	1.0	1,974	0.87	213	
Example 104	1.0	1,773	0.88	210	
Example 105	1.1	1,778	0.87	205	
Example 106	1.0	1,978	0.88	228	
Example 107	1.1	1,993	0.87	221	
Example 108	1.0	1,784	0.89	215	
Example 109	1.0	980	0.89	188	
Example 110	1.0	915	0.89	186	
Comparative Example 48	1.0	1,973	0.86	186	
Comparative Example 49	1.1	1,968	0.85	183	
Comparative Example 50	1.1	1,965	0.84	153	

#### Table 26 (continued)

5		<b></b>			
5	Examples	Properties of			
	and	magnetic recording medium			
	Comparative	Surface	Young's	Linear	Surface
10	Examples	roughness	modulus	absorption	resistivity
		Ra	(relative	$(\mu m^{-1})$	$(\Omega/ exttt{sq})$
		(nm)	value)		
15	Example 100	6.2	126	1.88	$6.1 \times 10^8$
	Example 101	6.5	124	2.14	$3.6 \times 10^{8}$
20	Example 102	6.6	127	1.86	$8.9 \times 10^{8}$
	Example 103	7.4	123	1.64	$1.8 \times 10^{9}$
	Example 104	7.0	129	2.68	$3.1 \times 10^8$
25	Example 105	7.6	125	2.65	1.6 x 10 <sup>8</sup>
	Example 106	6.2	125	2.22	$3.2 \times 10^9$
30	Example 107	6.4	130	2.63	$2.2 \times 10^{8}$
	Example 108	6.6	125	2.18	$4.6 \times 10^9$
	Example 109	7.5	134	1.94	$6.8 \times 10^9$
35	Example 110	7.0	125	1.52	8.9 × 10 <sup>9</sup>
	Comparative Example 48	9.1	124	1.18	$8.0 \times 10^{10}$
40	Comparative Example 49	11.6	119	1.24	$3.1 \times 10^{11}$
	Comparative Example 50	16.8	114	1.32	3.2 x 10 <sup>10</sup>

**Claims** 

45

50

- 1. A magnetic recording medium comprising:
  - (a) a non-magnetic base film;
  - (b) a non-magnetic undercoat layer on said non-magnetic base film, comprising a binder resin and non-magnetic acicular black iron-based composite particles; and
  - (c) a magnetic coating film comprising a binder resin and magnetic particles, comprising:
  - acicular hematite or iron oxide hydroxide particles having an average major axis diameter of from 0.02 to

0.30 µm,

5

10

20

25

30

35

- a coating layer on the surface of said acicular hematite or iron oxide hydroxide particles, comprising at least one organosilicon compound selected from:
  - (1) organosilane compounds obtainable by drying or heat-treating alkoxysilane compounds,
  - (2) polysiloxanes or modified polysiloxanes, and
  - (3) fluoroalkyl organosilane compounds obtainable by drying or heat-treating fluoroalkylsilane compounds, and
- from 1 to 20 parts by weight, per 100 parts by weight of said acicular hematite or iron oxide hydroxide particles, of carbon black particles which have a particle size of from 0.002 to 0.05 μm and which are adhered to at least a part of said coating layer.
- 2. A magnetic recording medium according to claim 1 wherein said alkoxysilane compound is represented by the general formula (I):

$$R_a^1SiX_{4-a}$$
 (I)

- wherein R<sup>1</sup> is  $C_6H_5$ -,  $(CH_3)_2CHCH_2$  or  $n-C_bH_{2b+1}$  (wherein b is an integer of from 1 to 18); X is  $CH_3O$  or  $C_2H_5O$ -; and a is an integer of from 0 to 3.
- 3. A magnetic recording medium according to claim 2, wherein said alkoxysilane compound is methyl triethoxysilane, dimethyl diethoxysilane, tetraethoxysilane, phenyl triethoxysilane, diphenyl diethoxysilane, methyl trimethoxysilane, dimethyl dimethoxysilane, tetramethoxysilane, phenyl trimethoxysilane, diphenyl dimethoxysilane, isobutyl trimethoxysilane or decyl trimethoxysilane.
- **4.** A magnetic recording medium according to any one of the preceding claims, wherein said polysiloxanes are represented by the general formula (II):

wherein R<sup>2</sup> is H- or CH<sub>3</sub>- and d is an integer of from 15 to 450.

- **5.** A magnetic recording medium according to claim 4, wherein said polysiloxanes are ones having methyl hydrogen siloxane units.
  - **6.** A magnetic recording medium according to any preceding claims, wherein said modified polysiloxanes are selected from:
    - (A) polysiloxanes modified with at least one polyether and/or polyester and/or epoxy compound, and
    - (B) polysiloxanes whose molecular terminal is modified with at least one group selected from carboxylic acid, alcohol and hydroxyl groups.
- 7. A magnetic recording medium according to claim 6, wherein said polysiloxanes (A) are represented by the general formula (III), (IV) or (V):

55

wherein  $R^3$  is -(-CH<sub>2</sub>-)<sub>h</sub>-;  $R^4$  is -(-CH<sub>2</sub>-)<sub>i</sub>-CH<sub>3</sub>;  $R^5$  is -OH, -COOH, -CH=CH<sub>2</sub>, -C(CH<sub>3</sub>)=CH<sub>2</sub> or -(-CH<sub>2</sub>-)<sub>j</sub>-CH<sub>3</sub>;  $R^6$  is -(-CH<sub>2</sub>-)<sub>k</sub>-CH<sub>3</sub>; g and h are each independently an integer of from 1 to 15; i, j and k are each independently an integer of from 0 to 15; e is an integer of from 1 to 50; and f is an integer of 1 from to 300;

wherein  $R^7$ ,  $R^8$  and  $R^9$  are -(-CH<sub>2</sub>-)<sub>q</sub>- and may be the same or different;  $R^{10}$  is -OH, -COOH, -CH=CH<sub>2</sub>, -C(CH<sub>3</sub>)=CH<sub>2</sub> or -(-CH<sub>2</sub>-)<sub>r</sub>-CH<sub>3</sub>;  $R^{11}$  is -(-CH<sub>2</sub>-)<sub>s</sub>-CH<sub>3</sub>; n and q are each independently an integer of from 1 to 15; r and s are each independently an integer of from 0 to 15; e' is an integer of from 1 to 50; and f' is an integer of from 1 to 300; or

$$CH_{3} - \stackrel{CH_{3}}{\stackrel{}{\stackrel{}}{\stackrel{}}} \stackrel{CH_{3}}{\stackrel{}{\stackrel{}}} \stackrel{CH_{3}}{\stackrel{}{\stackrel{}}} \stackrel{CH_{3}}{\stackrel{}{\stackrel{}}} \stackrel{CH_{3}}{\stackrel{}{\stackrel{}}} \stackrel{CH_{3}}{\stackrel{}{\stackrel{}}} \stackrel{CH_{3}}{\stackrel{}} {3}}{\stackrel{}} \stackrel{CH_{3}}{\stackrel{}} \stackrel{CH_{3}}{\stackrel{}} \stackrel{CH_{3}}{\stackrel{CH_{3}} \stackrel{CH_{3}}{\stackrel{}} \stackrel{CH_{3}}{\stackrel{CH_{3}} \stackrel{CH_{3}}{\stackrel{CH_{3}} \stackrel{CH_{3}}{\stackrel{CH_{3}} \stackrel{CH_{3}}{\stackrel{CH_{3}} \stackrel{CH_{3}}{\stackrel{CH_{3}} \stackrel{CH_{3}}{\stackrel{CH_{3}} \stackrel{CH_{3}}{\stackrel{CH_{3}} \stackrel{CH_{3}} \stackrel{CH_{3}}{\stackrel{CH_{3}} \stackrel{CH_{3}} \stackrel{CH_$$

wherein  $R^{12}$  is -(-CH<sub>2</sub>-)<sub>v</sub>-; v is an integer of from 1 to 15; t is an integer of from 1 to 50; and u is an integer of from 1 to 300.

**8.** A magnetic recording medium according to claim 6 or 7, wherein said polysiloxanes (B) are represented by the general formula (VI):

wherein  $R^{13}$  and  $R^{14}$  are -OH,  $R^{16}$ OH or  $R^{17}$ COOH and may be the same or different;  $R^{15}$  is -CH<sub>3</sub> or -C<sub>6</sub>H<sub>5</sub>;  $R^{16}$  and  $R^{17}$  are -(-CH<sub>2</sub>-)<sub>y</sub>-; y is an integer of from 1 to 15; w is an integer of from 1 to 200; and x is an integer of from 0 to 100.

A magnetic recording medium according to any one of the preceding claims, wherein said fluoroalkylsilane compounds are represented by the general formula (VII):

$$CF_3(CF_2)_zCH_2CH_2(R^{18})_aSiX_{4-a'}$$
 (VII)

wherein  $R^{18}$  is  $CH_{3-}$ ,  $C_{2}H_{5-}$ ,  $CH_{3}O$ - or  $C_{2}H_{5}O$ -; X is  $CH_{3}O$ - or  $C_{2}H_{5}O$ -; and z is an integer of from 0 to 15; and a' is an integer of from 0 to 3.

- 10. A magnetic recording medium according to any one of the preceding claims, wherein said acicular hematite or iron 10 oxide hydroxide particles are acicular manganese-containing hematite or goethite particles.
  - 11. A magnetic recording medium according to any one of the preceding claims, wherein the amount of said coating organosilicon compounds is from 0.02 to 5.0 % by weight, calculated as Si, based on the total weight of the organosilicon compounds and said acicular hematite or iron oxide hydroxide particles.

12. A magnetic recording medium according to any one of the preceding claims, wherein said acicular hematite or iron oxide hydroxide particles are particles having a coat on at least a part of the surface thereof comprising at least one hydroxide or oxide selected from hydroxides of aluminum, oxides of aluminum, hydroxides of silicon and oxides of silicon in an amount of from 0.01 to 50 % by weight, calculated as Al and/or SiO2, based on the total weight of the acicular hematite or iron oxide hydroxide particles.

- 13. A magnetic recording medium according to any one of claims 1 to 11, which has a coating film gloss of from 122 to 300 %, a coating film surface roughness Ra of not more than 12.0 nm, a coating film linear absorption of from 1.10 to 2.00  $\mu m^{-1}$  and a surface resistivity of not more than 5 x 10<sup>8</sup>  $\Omega$ /sq.
- 14. A magnetic recording medium according to claim 12, which has a coating film gloss of from 124 to 300 %, a coating film surface roughness Ra of not more than 12.0 nm, a coating film linear absorption of from 1.10 to 2.00 µm<sup>-1</sup> and a surface resistivity of not more than  $5 \times 10^8 \Omega/\text{sq}$ .
- 15. A non-magnetic substrate comprising:

a non-magnetic base film; and a non-magnetic undercoat layer as defined in any one of claims 1 to 12.

- 16. A non-magnetic substrate according to claim 15, which has a coating film gloss of from 185 to 300 %, a coating film surface roughness Ra of from 0.5 to 10.0 nm and a surface resistivity of not more than 5 x  $10^9 \Omega$ /sq.
  - 17. A non-magnetic substrate according to claim 16, wherein the gloss is from 187 to 300 % and the surface roughness Ra is from 0.5 to 9.5 nm.

40

5

15

20

25

50

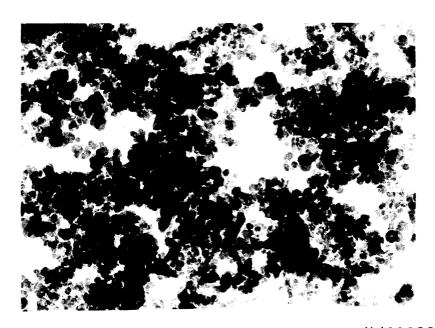
45

FIG.1



(×60000)

FIG.2



(×60000)

FIG.3



(×60000)

FIG.4



(×60000)